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# Solid Waste: Methodology for Evaluation
## Volume II: Analysis of Residual Wastes

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This report presents the results of a program of evaluation of analytical methods for "Total Content" of residual wastes. Candidate methods were first studied and modified, and then evaluated by analyzing a variety of industrial residuals for a broad range of organic compounds and metals. The results, based on statistical analysis of over 10,000 data points, are quoted primarily in terms of observed accuracy and precision. The methods depend on extraction, gel permeation chromatography cleanup, preconcentration, and GC/MS analysis for identification and quantitation of semivolatile organics; purge-and-trap and GC/MS for purgeable organics; and acid digestion combined with AAS or ICP determinations for metals. An edited text of the evaluated methods is presented in the format specified by EMSL-Cincinnati for standard methods as an appendix to this report.

Southern Research Institute and Battelle-Columbus Laboratories exchanged methodologies developed for Total Content and Potential Mobility and applied these procedures to the analyses of samples in an interlaboratory study. Results of SoRI's contribution to the interlaboratory study are presented in the appendices to this report.

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1981

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# SECTION 1

# INTRODUCTION

Recent legislation, specifically the Resource Conservation and Recovery Act of 1976 (RCRA) has required the Office of Solid Waste (OSW) within EPA's Office of Water and Waste Management (OWWM) to identify waste materials that may be hazardous and to provide methodology for characterizing these wastes. As part of OSW's continuing program to develop methodology for analyzing industrial residuals and for determining the potential mobility of toxicants in wastes, the Effluent Guidelines Division (EGD), also within OWWM, has provided technical and administrative support to OSW through existing contracts for the development of analytical methodologies.

Southern Research Institute (SoRI), under Technical Directive No. 108 of EPA Contract No. 68-02-2685, was tasked with the responsibility of developing analytical methods for total content of organic and inorganic species in industrial residuals and Battelle-Columbus Laboratories (BCL) was given responsibility for the potential mobility (leachate) methods. Specifically, SoRI was required to develop the methods for total content by evaluating and modifying, as necessary, existing methods from several sources, including Midwest Research Institute, Battelle-Columbus Laboratories, Department of Environmental Health of the University of Washington, and EPA-Cincinnati. Following this initial evaluation phase of the study, a more extensive evaluation program was undertaken with the most promising methods by analyzing a broad range of industrial residuals. The compounds that were determined in the wastes represented a wide range of toxic pollutant classes expected to be encountered in the hazardous wastes program. The analytical and statistical data from the evaluation study for the proposed methodology for total content are given in Appendices I and IV.

After evaluation of the methods, SoRI and BCL exchanged the methodologies developed for total content and potential mobility and applied the procedures to the analysis of nominally identical samples in an interlaboratory study (the data resulting from SoRI's participation in this study are presented in Appendices I, II and III).

The goals for the developed methods were that they would be:

- technically sound;
- feasible and practical to implement;
- developed within known and acceptable limits for accuracy and precision;
- within the capability of a production-type laboratory;
- similar in scope to EPA wastewater methods embodying GC/MS (e.g., 624 and 625) and AAS or ICP;

- implemented at a cost not to exceed $2000 per sample analysis when performed routinely;

- presented in the format specified by EMSL-Cincinnati for standard methods.

Results of the various phases of this project are presented in the following sections of this report. The comparison of the data from the interlaboratory study for total content and potential mobility are presented by BCL in the final report, Task 2013, Contract No. 68-03-2552.

# SECTION 2

## CONCLUSIONS

The primary conclusions drawn from this study were the following:

- Overall, it was concluded that the three candidate methods that were evaluated can be applied with confidence to the survey analysis of a relatively broad range of industrial residuals. This conclusion was based on the statistical analysis of over 10,000 data points which were expressed primarily in terms of observed accuracy and precision.

- The methods appear to be sound technically and practical in their implementation. It is estimated that the analyses can be performed by a production-type laboratory utilizing appropriately supervised technicians and junior-level professionals for sample preparation and upper-level professionals for the supervision of GC/MS analysis and data interpretation. The cost of a complete analysis by the proposed methods appears to be well within the goal of $2000 per sample (at current typical rates) with a sample turnaround time comparable to that experienced with similar analytical methodology for wastewater.

- Based upon the recoveries of known amounts of compounds spiked into seven sample matrices, the methods can be expected to yield, in general, results whose accuracy will be in the range of approximately -50 to 150% of the true values.

- Results of replicate analyses indicate that the methods should be capable of yielding data with a precision in the range of 10 to 25% relative standard deviation (RSD) for organics and 3 to 7% RSD for metals.

- Perhaps the greatest single limitation to the usefulness of data resulting from the proposed methods lies in the practical difficulties associated with the collection of a truly representative sample of the extremely complex matrices that are typical of residual wastes. Indeed, isolation of a representative aliquot from a larger sample in the laboratory has posed substantial problems in some instances. It is probable that poor sample homogeneity and inadequate aliquoting in the laboratory contribute substantially to the variability observed in the analytical results.

# SECTION 3

# RECOMMENDATIONS

The three methods described in Appendix V of this report are applicable to the determination of various organic compounds and elements in residual wastes. These methods depend on extraction, gel permeation chromatographic cleanup, preconcentration, and GC/MS analysis for identification and quantitation of semivolatile organics; purge-and-trap and GC/MS for purgeable organics; and acid digestion combined with AAS determination for metals. Because of the potential complexity of wastes, these methods are broad in scope. Of course, no single method is applicable to all wastes and some modifications (e.g., change in extraction procedures and cleanup chromatography before final analysis) may be necessary for unusual waste types.

## PURGEABLE AND SEMIVOLATILE ORGANICS

Although 78 organic compounds were determined in the evaluation of the proposed methods, there are many other compounds that may need to be determined in wastes. The nature of the waste will dictate what is to be determined. For example, compounds such as dioxin, and mixtures such as toxaphene and PCB's may need to be determined in a specific residual waste. The results of this study should be helpful both in ascertaining the appropriateness of these methods for the analysis of the specific compounds studied as well as the much wider spectrum of compounds expected to be found in wastes.

When a sample is submitted for analysis, a general statement should be made with respect to solids content and organic or inorganic content. If these data are not available, they should be determined in the laboratory prior to further workup of the sample. This information will help determine the sample size and the manner in which the sample will be prepared for analysis. For example, those samples that are predominantly aqueous are extracted with solvent after pH adjustment without the addition of dilution water to the sample. Conversely, water is added and the pH adjustment made prior to the initial extraction step when the sample is predominantly an organic liquid. In all situations, attempts were made to use equal volumes of the aqueous sample and the organic extracting solvent. The organic or inorganic content will dictate the amount of sample taken for analysis. Also, if concentrations of purgeable organics are relatively high, a pentane or carbon disulfide extraction procedure may be of use as an alternative to the purge-and-trap procedure.

Because of the wide range of compounds the analyst may encounter in an industrial residual, a limitation of compounds to be screened should be established. One way this may be done is through an engineering assessment of the process producing the residual waste. Also, those compounds not having a significant environmental impact may be eliminated from the analysis regimen, or the analytical program may be narrowed in scope by only identifying and quantitating the major peaks found in the GC/MS chromatograms.

Two areas that merit additional evaluation are the liquid-liquid extraction procedure for semivolatile organics and the capillary GC/MS analysis for organic compounds. When a residual waste sample is free of basic compounds, e.g., benzidine and 3,3'-dichlorobenzidine, it may be possible that sample preparation time can be substantially reduced by performing an acid/neutral extraction, thus eliminating several steps in the sample preparation. Capillary GC/MS analyses may have sufficient resolution to differentiate between certain isomeric compounds. For example, the packed-column GC retention times and mass spectra of the pairs anthracene and phenanthrene, chrysene and benzo(a)anthracene, and benzo(b)fluoranthene and benzo(k)fluoranthene are not sufficiently different to make an unambiguous distinction between these compounds. In such situations, capillary GC/MS or HPLC analyses may be preferred. Also, the utility of a single extraction followed by capillary GC/MS should be evaluated.

## METALS

The University of Washington procedure was selected as the method of choice for digesting and solubilizing the metals in a variety of waste types ranging from inorganic solids to samples of high organic content. Concern over potentially hazardous conditions resulting from the oxidation of organic material in a sealed glass vial appeared to be unfounded. Consultation with researchers at the University of Washington likewise indicated that they had encountered no problems during the digestion of many types of samples in their laboratories.

Despite the fact that only a selected group of more toxic metals were determined in this study, it seems reasonable to assume that the digestion procedure should be equally effective for numerous other elements. Furthermore, the digested samples may be amenable to analysis by inductively coupled plasma emission spectroscopy (ICP) as well as the atomic adsorption spectroscopy (AAS) procedures used in this study.

Although the digestion procedure is considered to be applicable to a wide range of industrial wastes, it is recognized that low recoveries of some elements may be experienced in certain types of waste materials. Therefore, it is imperative that each matrix be evaluated for interference effects by determining the recovery efficiency for spikes added to the samples prior to the digestion step.

Data obtained during this study suggest that recovery problems are more likely to occur with the following elements: antimony, arsenic, selenium and silver. In general, spikes of these metals, added to samples prior to digestion, resulted in poor recovery efficiencies. The same level of spikes added to digested, filtered samples, however, gave essentially complete spike recovery. On the basis of these data it seems likely that significant losses of analyte occurred during the filtration step. The two most plausible explanations for analyte loss are (1) the precipitation of the elements as insoluble compounds and (2) the adsorption, occlusion, or complexation of the analyte ions by the undissolved sample residue. Unfortunately, constraints in both time and money precluded any investigative studies to resolve this problem. Because of the relative toxicity of the elements in question and their potential impact on the environment, it is recommended that additional studies be conducted in order to develop adequate analytical capability for their determination in residual wastes.

# SECTION 4

## SELECTION AND MODIFICATION OF METHODS

Several methods for the determination of purgeable organics, semivolatile organics, and metals in residual wastes are available for consideration. These methods were reviewed, modifications were proposed, and candidate methods were then evaluated with various types of waste materials. Summaries of the procedures with modifications are presented in Tables 1 through 12. The rationale for selection and modification is discussed in the following three sections that represent three major classifications of compounds and elements to be determined in residual wastes. Priority pollutant compounds were only used in this study because of their availability and historical background. The methods described below are to be applied to those organic compounds and metals found in industrial waste.

### PURGEABLE ORGANICS

Four methods for the determination of purgeable organics in residual wastes were selected for evaluation based on recommendations of Effluent Guidelines Division: Midwest Research Institute (MRI),[1] University of Washington (UW),[2] Environmental Protection Agency-Cincinnati (EPA),[3] and Environmental Protection Agency Interim Method (EPA-IM).[4]

All methods were similar to the extent that an aliquot of waste material was purged of volatile organics with an inert gas in a specially designed purging chamber. The volatilized organic compounds were then collected in a sorbent trap. Following the purge cycle, the sorbent trap was heated and backflushed to desorb the purgeable organics into the inlet of a GC/MS system.

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1. "Development of Analytical Test Procedures for the Measurement of Organic Priority Pollutants in Sludge and Sediments," Midwest Research Institute, Final Report EPA Contract No. 68-03-2695, June 26, 1979.

2. "Presence of Priority Pollutants in Sewage and their Removal on Sewage Treatment Plants," University of Washington, Annual Report Grant R.806102, July 31, 1979.

3. "Method for Purgeable Organics," (Unpublished), Personal Communication with MERL-EPA, Cincinnati, Ohio 45268, March 1980.

4. "Interim Methods for the Measurement of Organic Priority Pollutants in Sludges," U. S. Environmental Protection Agency, Environmental Monitoring and Support Laboratory, Cincinnati, Ohio 45268, September 1979.

TABLE 1. MIDWEST RESEARCH INSTITUTE PROCEDURE FOR PURGEABLE ORGANICS

| Analysis scheme | Procedure | Modifications | Problems/comments |
|---|---|---|---|
| Purge device | Tekmar Liquid Sample Concentrator Model LSC-1. | | |
| Trap column | 3% OV-1 on Chromosorb-W, 100/110 mesh; Tenax-GC, 60/80 mesh; Silica gel—Davison Grade 15, 35/60 mesh; Coconut charcoal—Barnebey-Cheney, 26 mesh. | 10¼ in. x 0.105 ID stainless steel tubing packed with 4 in. of Tenax, 60/80 mesh and 2 in. of Silica gel, Davison Grade 15, 35/60 mesh. | The 3% OV-1 and the silica gel are special order items. Since extract amounts of the packing materials are not indicated for best desorption efficiency, commercial volatile trap columns that are EPA recommended (i.e., Supelco) should be used. If charcoal is desired, then commercially bought traps can be modified. |
| GC column | 2.4-2.8 m x 1/8 in. OD stainless steel or x 2 mm ID glass column packed with 0.2% Carbowax 1500 on Carbopack C, 80/100 mesh. | 1.8 m x 2 mm ID glass column packed with 1% SP-1000 on Carbopack B, 60/80 mesh. | SP-1000 on Carbopack B packing has a higher degree of inertness shown by no tailing of the methanol solvent peak. Solvent tailing can interfere with early peaks. |
| MS calibration compound | p-Bromofluorobenzene. Not a direct injection.* | p-Bromofluorobenzene, 20 ng. Direct injection. | If purging device is connected directly to GC via injection port, then purging the p-bromofluorobenzene would prove more efficient. |

* Analyze organic-free water with 75 ng of p-bromofluorobenzene + surrogate standards and internal standards to meet abundance criteria.

TABLE 1. MIDWEST RESEARCH INSTITUTE PROCEDURE FOR PURGEABLE ORGANICS (CONTINUED)

| Analysis scheme | Procedure | Modifications | Problems/comments |
|---|---|---|---|
| Mass spectrometer | Finnigan or equivalent. | Hewlett-Packard 5985A or equivalent. | |
| Standards | Supelco mixture or neat compounds prepared daily; 10 mL of 5 μg/L and 25 μg/L standards. | Supelco purgeables A and B standard mixtures. | Premix multiple standards (i.e., Supelco). Allow for case of spiking as well as reducing solvent volume being introduced into the sample. This lessens the chance of interference with any early peaks (i.e., methylene chloride). |
| Internal standards | 1000 μg/50 mL with 9-μL injections; bromochloromethane; 2-bromo-1-chloropropane; 1,4-dichlorobutane. Prepare weekly. | Deuterated toluene. | The three multiple component standard would best serve as surrogate standards to monitor the sample through analysis. |
| Purge conditions | 12 min purge at ∿25 °C. Desorb 3 min at 180 to 200 °C. | 12-min purge at room temperature. Desorb 4 min at 180 °C. Helium flow 40 mL/min. | |
| GC conditions | 60 °C (3 min), 8 °C/min to 160 °C (hold). Flow—30 mL/min. | 50 °C (4 min), 10 °C/min to 225 °C (20 min). Flow—40 mL/min. | |
| MS conditions | 20-275 a.m.u., 3 to 5 sec/scan. | 45-345 a.m.u., 2 to 3 sec/scan. | |
| Sample size | 10 mL. | 10 mL containing a minimum of 50 mg of solid content. | |

TABLE 1. MIDWEST RESEARCH INSTITUTE PROCEDURE FOR PURGEABLE ORGANICS (CONTINUED)

| Analysis scheme | Procedure | Modifications | Problems/comments |
|---|---|---|---|
| Solids determination | Weigh two 1-mL aliquots before and after drying at 110 °C. | | |
| Sample handling | Pipet amount of sludge which contains 50 mg of dry solid and dilute to 10 mL. | | |
| Spiking | Weigh vial empty and full, spike. Put on roller mill 16 h at 4 °C with 1- or 2-1/8 in. ball bearings. | | |

TABLE 2. UNIVERSITY OF WASHINGTON PROCEDURE FOR PURGEABLE ORGANICS

| Analysis scheme | Procedure | Modifications | Problems/comments |
|---|---|---|---|
| Purge device | 1000-mL Erlenmeyer flask and purging headpiece with 24/40 standard joints. | Tekmar Liquid Sample Concentrator Model LSC-1. | |
| Trap column | 220 mm x 6.35 mm OD stainless steel tubing packed with 0.4 g Tenax GC and 0.2 g Chromosorb 102; 120 mm x 6.35 mm OD stainless steel trap guard packed with 0.2 g Tenax GC. | 10¼ in. x 0.105 ID stainless steel tubing packed with 4 in. of Tenax, 60/80 mesh and 2 in. of Silica gel, Davison Grade 15, 35/60 mesh. | |
| GC column | 12 ft x 2 mm ID glass column packed with 0.2% Carbowax 1500 on Carbopack C, 60/80 mesh. | 1.8 m x 2 mm ID on-column injection column packed with 1% SP-1000 on Carbopack B, 60/80 mesh. | SP-1000 on Carbopack B packing has a high degree of inertness shown by no tailing of the methanol solvent peak. Solvent tailing can interfere with early peaks. |
| MS calibration compound | Not listed in method. | p-Bromofluorobenzene, 20 ng. Direct injection. | |
| Mass spectrometer | Not listed in method. | Hewlett-Packard 5985. | |
| Standards | No protocol listed in method. | Supelco's purgeables A and B standard mixtures. | Premix multiple standards (i.e., Supelco). Allows for ease of spiking as well as reducing solvent volume being introduced into the sample and lessens the chance of interference with any early peaks. |

TABLE 2. UNIVERSITY OF WASHINGTON PROCEDURE FOR PURGEABLE ORGANICS (CONTINUED)

| Analysis scheme | Procedure | Modifications | Problems/comments |
|---|---|---|---|
| Internal standards | 2-Bromo-1-chloropropane. | Deuterated toluene. | 2-Bromo-1-chloropropane is difficult to find except as one of three compounds in the internal standard mixture from Supelco. |
| Purge conditions | Purge under stirring for 20 min in water bath 60 °C. Desorb 250 °C for 4 min. Nitrogen flow 200 mL/min. | 12-min purge at room temperature. Desorb 4 min at 180 °C. Helium flow 40 mL/min. | |
| GC conditions | 40 °C, 8 °C/min to 200 °C (hold for 16 min). | 50 °C (4 min), 10 °C/min to 225 °C (20 min). Flow—40 mL/min. | Maximum temperature of UW program is 200 °C for 16 min; yet the column packing has a maximum temperature of 175 °C. |
| MS conditions | 34 to 334 a.m.u. (no scan time given). | 45-345 a.m.u., 2 to 3 sec/scan. | |
| Sample size | Sample diluted up to 100 mL but NO amount was indicated in method. | 10 mL containing a minimum of 50 mg of solid content. | |
| Solid determination | Use standard method gravimetric. | | |
| Sample handling | Sludge dilution at 7 to 25% sludge, 25 to 40% sewage. | | |
| Spiking | 1 mg/mL (10 μL) in 50 mL water organic free = 2 ng/μL. | | |

TABLE 3. ENVIRONMENTAL PROTECTION AGENCY PROCEDURE FOR PURGEABLE ORGANICS

| Analysis scheme | Procedure | Modifications | Problems/comments |
|---|---|---|---|
| Purge device | Tekmar Liquid Sample Concentrator Model LSC-1 (or hand rigged). | | |
| Trap column | 3% OV-1 on Chromosorb-W, 60/80 mesh; Tenax-GC, 60/80 mesh; Silica gel—Davison Grade 15, 35/60 mesh or equivalent; Coconut charcoal—Barnebey-Cheney, 26 mesh or equivalent. | 10½ in. x 0.105 ID stainless steel tubing packed with 4 in. of Tenax, 60/80 mesh and 2 in. of Silica gel, Davison Grade 15, 35/60 mesh. | Commercial sources of traps can be packed in the laboratory from special order packing. |
| GC column | 8 ft x 2 mm ID glass column packed with 0.2% Carbowax 1500 on Carbopack C, 60/80 mesh. Flow 25 mL/min. | 1.8 m x 2 mm ID on-column injection column packed with 1% SP-1000 on Carbopack B, 60/80 mesh. Flow 60 mL/min. | SP-1000 on Carbopack B packing has a higher degree of inertness shown by no tailing of the methanol solvent peak. Solvent tailing can interfere with early peaks. |
| MS calibration compound | Pentafluorobromobenzene. | p-Bromofluorobenzene, 20 ng. Direct injection. | Pentafluorobromobenzene is a difficult compound to locate. p-Bromofluorobenzene is less expensive and a more available compound for the calibration of MS. |
| Mass spectrometer | Finnigan 1015 series or equivalent. | Hewlett-Packard 5985. | |

TABLE 3. ENVIRONMENTAL PROTECTION AGENCY PROCEDURE FOR PURGEABLE ORGANICS (CONTINUED)

| Analysis scheme | Procedure | Modifications | Problems/comments |
|---|---|---|---|
| Standards | Complex test mixture—100 ng/µL; "quality check" sample 20 µg/L (made from 100 ng/µL). | Supelco's purgeable A and B standard mixtures. | Premix multiple standards (i.e., Supelco). Allows for ease of spiking as well as reducing solvent volume being introduced into the sample. This lessens the chance of interference with any early peaks. |
| Internal standards | Method recovery spiking solution. | Deuterated toluene. | |
| Purge conditions | 12 min purge at room temperature. Desorb trap at 180 to 200 °C for 3 min. Nitrogen or helium flow 40 mL/min. | 12-min purge at room temperature. Desorb 4 min at 180 °C. Helium flow 40 mL/min. | |
| GC conditions | 60 °C (3 min), 8 °C/min to 160 °C (hold until all compounds are off). | 50 °C (4 min), 10 °C/min to 225 °C (20 min). Flow—40 mL/min. | |
| MS conditions | Not given in this procedure. | 45-345 a.m.u., 2 to 3 sec/scan. | |
| Sample size | 10 mL. | 10 mL containing a minimum of 50 mg of solid content. | |
| Solid determination | Gravimetric. | | |

TABLE 3. ENVIRONMENTAL PROTECTION AGENCY PROCEDURE FOR PURGEABLE ORGANICS (CONTINUED)

| Analysis scheme | Procedure | Modifications | Problems/comments |
|---|---|---|---|
| Sample handling | Pipet amount of sludge which contains 50 mg of dry solids and dilute to 10 mL. | | |
| Spiking | Spike at two times concentration in samples or ten times the lower limit of detection. Place sample on roller mill at 4 °C for 16 h to mix. | | |

TABLE 4. ENVIRONMENTAL PROTECTION AGENCY INTERIM METHOD FOR PURGEABLE ORGANICS (SEPTEMBER 1979)

| Analysis scheme | Procedure | Modifications | Problems/comments |
|---|---|---|---|
| Purge device | Tekmar Liquid Sample Concentrator Model LSC-1. | | |
| Trap column | 3% OV-1 on Chromosorb-W, 60/80 mesh; Tenax-GC, 60/80 mesh; Silica gel—Davison Grade 15, 35/60 mesh; Coconut charcoal—Barnebey-Cheney, 26 mesh. | 10¼ inch x 0.105 ID stainless steel tubing packed with 4 inches of Tenax, 60/80 mesh and 2 inches of Silica gel, Davison Grade 15, 35/60 mesh. | Commercial sources of traps can be packed in the laboratory from special order packing. |
| GC column | 8 ft x 0.1 in stainless steel or a glass column packed with 0.2% Carbowax 1500 on Carbopack C, 60/80 mesh. | 1.8 m x 2 mm ID glass on-column injection column packed with 1% SP-1000 on Carbopack B, 60/80 mesh. | SP-1000 on Carbopack B packing has a high degree of inertness shown by no tailing of the methanol solvent peak. Solvent tailing can cause GC interference with early peaks (i.e., methylene chloride). |
| MS calibration compound | p-Bromofluorobenzene, 75 ng. | p-Bromofluorobenzene, 20 ng. Direct injection. | |
| Mass spectrometer | Finnigan 4000 or equivalent. | Hewlett-Packard 5985. | |
| Standards | Made from neat stock prepared daily; 5 μg/L and 25 μg/L standard. | Supelco purgeables A and B standard mixtures. | Standards made by the method are time consuming and introduce many places for human error. |

TABLE 4. ENVIRONMENTAL PROTECTION AGENCY INTERIM METHOD FOR PURGEABLE ORGANICS (SEPTEMBER 1979) (CONTINUED)

| Analysis scheme | Procedure | Modifications | Problems/comments |
|---|---|---|---|
| Internal standards | Bromochloromethane; 2-bromo-1-chloropropane; 1,4-dichlorobutane. Made from neat standard so to use 9-µL injections. | Deuterated toluene. | 2-Bromo-1-chloropropane is not easily found as a single compound. |
| Purge conditions | 12 min purge at room temperature. Desorb 3 min. Nitrogen or helium flow 40 mL/min. | 12-min purge at room temperature. Desorb 4 min at 180 °C. Helium flow 40 mL/min. | |
| GC conditions | 60 °C (3 min), 8 °C/min to 160 °C (hold until all compounds come off). | 50 °C (4 min), 10 °C/min to 225 °C (20 min). Flow—40 mL/min. | |
| MS conditions | 20-275 a.m.u., 3 to 5 sec/scan. | 45-345 a.m.u. 5 sec/scan. | |
| Sample size | 50 mg solid content diluted up to 10 mL. | 10 mL containing a minimum of 50 mg of solid content. | |
| Solid determination | Gravimetric. | | |
| Sample handling | Pipet amount of sludge which contains 50 mg of dry solid and dilute to 10 mL. | | |
| Spiking | Day 1 spiking—two times concentration found or ten times lower limit. | | |

TABLE 5. MIDWEST RESEARCH INSTITUTE PROCEDURE FOR SEMIVOLATILE ORGANICS

| Analysis scheme | Procedure | Modifications | Problems/comments |
|---|---|---|---|
| Sludge | Homogenize sludge 1 min. Transfer four 80-mL aliquots to 250-mL centrifuge tubes. | Transfer one aliquot (10-100 mL) to a 250-mL centrifuge tube. | If the amount of suspended solids is low, then separation and extraction will not be difficult. Thus, a single but larger aliquot of sludge may be extracted. |
| Adjust to pH $\geq$ 11 | Adjust pH with 6 N NaOH. Homogenize briefly. | Adjust pH with 10 N NaOH. | With a stronger base, less volume is required to adjust the pH of the sludge. |
| | | | If the amount of suspended solids is high, then the homogenizer is difficult to clean. Thus, the total volume in the centrifuge tube may approach maximum capacity or a loss of some of the organic priority pollutants may occur. |
| Extract 3X | Add 80 mL of $CH_2Cl_2$. Homogenize for 60 sec. Centrifuge the samples at 3000 rpm for 30 min. Withdraw extracts with a 100-mL pipette and transfer to a 500-mL separatory funnel. Extract sample two more times. | Add 100 mL of $CH_2Cl_2$ Withdraw extract with a 100-mL glass syringe and transfer to a 300-mL fleaker. | If 100-mL addition not possible, add enough to fill to neck of centrifuge tube. Samples are centrifuged at 1400 relative centrifugal force. |

TABLE 5. MIDWEST RESEARCH INSTITUTE PROCEDURE FOR SEMIVOLATILE ORGANICS (CONTINUED)

| Analysis scheme | Procedure | Modifications | Problems/comments |
|---|---|---|---|
| Dry extract | Drain pooled $CH_2Cl_2$ extracts through column of $Na_2SO_4$ (100 mm x 20 mm) into K-D evaporator. Wash drying column with an additional 100 mL of $CH_2Cl_2$ and combine with Eluent extracts. Retain aqueous layer to be equally distributed to each of the four centrifuge tubes. | Drain pooled extracts through a drying tube of anhydrous $Na_2SO_4$ (60 mm x 17 mm) into K-D evaporator. Wash drying column with an additional 30 mL of $CH_2Cl_2$ into the K-D evaporator. Retain aqueous phase for pH-2 extraction. | |
| Concentrate base/ neutral extract | Concentrate the extract to 8 mL if viscous and darkly colored or 5 mL if not viscous or slightly colored. Transfer and dilute to 10 mL and store at 4 °C for GPC cleanup. | Concentrate the extract to 10 mL and immediately put entire amount or an aliquot through GPC cleanup. | |
| Cleanup | A 5-mL aliquot of concentrated extract is put through GPC cleanup (GPC Autoprep 1002 with a 25-mm ID column containing 50-60 g Bio-Beads S-X3). Elute with $CH_2Cl_2$ at 7-10 psi. Discard first 100 mL and collect next 150 mL in 500-mL brown bottles. Clean column with 100 mL of $CH_2Cl_2$. | Transfer total concentrate or an aliquot quantitatively to GPC column (450 mm x 19 mm column of Bio-Beads S-X3). Elute with $CH_2Cl_2$. Discard first fraction (∿65 mL) and collect the second fraction (∿120 mL). | |

TABLE 5. MIDWEST RESEARCH INSTITUTE PROCEDURE FOR SEMIVOLATILE ORGANICS (CONTINUED)

| Analysis scheme | Procedure | Modifications | Problems/comments |
|---|---|---|---|
| Concentrate cleaned extract | Concentrate cleaned extract to ∿10 mL with K-D evaporator and then to ∿3 mL with micro Snyder column. Transfer and dilute to 5 mL with $CH_2Cl_2$. | As extract elutes from S-X3 column, pass eluent through anhydrous $Na_2SO_4$ column into a K-D evaporator. Concentrate extract to ∿2 mL with K-D evaporator and then to ∿0.5 mL with micro Snyder column. Transfer and dilute to 1 mL with $CH_2Cl_2$. | This modification reduces the number of transfers and minimizes sample losses. |
| Analysis | GC/MS. | Store sample in 1-mL capped serum bottle at 4 °C for GC/MS analysis. | |
| Aqueous phase remaining after base/neutral extraction | Adjust pH $\geq 2$ with 6 $\underline{N}$ HCl. Homogenize briefly. | Shake by hand. | Care must be taken in this step due to excessive gas release and foaming. Two to five mL of acid are required to adjust pH of the sludges examined. If the amount of suspended solids is high, then the sample may have to be divided between two centrifuge tubes. |

TABLE 5. MIDWEST RESEARCH INSTITUTE PROCEDURE FOR SEMIVOLATILE ORGANICS (CONTINUED)

| Analysis | Procedure | Modifications | Problems/comments |
|---|---|---|---|
| Extract 3X | Add 80 mL of $CH_2Cl_2$. Homogenize for 60 sec. Centrifuge the samples at 3000 rpm for 30 sec. Withdraw extracts with a 100-mL pipette and transfer to a 500-mL separatory funnel. Extract sample two more times. | Add 50-80 mL of $CH_2Cl_2$. Shake by hand or homogenize. Withdraw extract with a 100-mL glass syringe and transfer to 300-mL fleaker. | $CH_2Cl_2$ is added to fill up to neck of centrifuge tube. For large amounts of suspended solids and small volumes of $CH_2Cl_2$, sample is shaken by hand to minimize foaming and spillage. Samples are centrifuged at 1400 R.C.F. |
| Dry extract | Drain pooled $CH_2Cl_2$ extracts through column of $Na_2SO_4$ (100 mm x 70 mm) into K-D evaporator. Wash drying column with an additional 100 mL of $CH_2Cl_2$ and combine with eluent extracts. | Drain pooled extracts through a drying tube of anhydrous $Na_2SO_4$ (60 mm x 17 mm) into K-D evaporator. Wash drying column with an additional 30 mL of $CH_2Cl_2$ into the K-D evaporator. | |
| Concentrate acid extract | Concentrate the extract to 8 mL if viscous and darkly colored or 5 mL if not viscous or slightly colored. Transfer and dilute to 10 mL and store at 4 °C for GPC cleanup. | Concentrate the extract to 5 mL and immediately put entire amount or aliquot through GPC cleanup. | |

TABLE 5. MIDWEST RESEARCH INSTITUTE PROCEDURE FOR SEMIVOLATILE ORGANICS (CONTINUED)

| Analysis scheme | Procedure | Modifications | Problems/comments |
|---|---|---|---|
| Cleanup | A 5-mL aliquot of concentrated extract is put through GPC cleanup (GPC Autoprep 1002 with a 25-mm ID column containing 50-60 g Bio-Bead S-X3). Elute with $CH_2Cl_2$ at 7-10 psi. Discard first 100 mL and collect next 150 mL in 500-mL brown bottles. Clean column with 100 mL of $CH_2Cl_2$. | Transfer total concentrate or an aliquot quantitatively to GPC column (∿450 mm x 19 mm column of Bio-Beads S-X3). Elute with $CH_2Cl_2$. Discard first fraction (∿65 mL) and collect the second fraction (∿120 mL). | |
| Concentrate cleaned extract | Concentrate cleaned extracts to ∿10 mL with K-D evaporator and then to ∿3 mL with micro Snyder column. Transfer and dilute to 5 mL with $CH_2Cl_2$. | As extract elutes from S-X3 column, pass eluent through anhydrous $Na_2SO_4$ column into a K-D evaporator. Concentrate extract to ∿2 mL with K-D evaporator and then to ∿0.5 mL with micro Snyder column. Transfer and dilute to 1 mL with $CH_2Cl_2$. | This modification reduces the number of transfers and minimizes sample losses. |
| Analysis | GC/MS. | Store sample in 1 mL capped serum bottle at 4 °C for GC/MS analysis. | |

TABLE 6. BATTELLE-COLUMBUS LABORATORIES PROCEDURE FOR SEMIVOLATILE ORGANICS

| Analysis scheme | Procedure | Modifications | Problems/comments |
|---|---|---|---|
| A. Neutral fraction | | | |
| Sludge | Homogenize sludge. Transfer 100 g into 200-mL centrifuge tube. | Transfer 100 mL into 250-mL centrifuge tube. | |
| Acidify | Acidify with 5 g of $KHSO_4$. | Homogenize for 30 sec. | Assures uniform mixing of the $KHSO_4$. More than 5 g $KHSO_4$ may be necessary. |
| Extract 3X | Add 100 mL of $CH_2Cl_2$. Homogenize for 1 min. Centrifuge. Remove extract with a 50-mL syringe and transfer to 500-mL round-bottom flask. Extract sample two more times. Combine extracts. | Homogenize for 30 sec. Centrifuge at 1400 R.C.F. for 30 min. Remove extract with a 100-mL glass syringe and transfer to a K-D evaporator. | |
| Concentrate extract | Concentrate extract to 60-80 mL on a rotating evaporator at 35 °C. | Concentrate extract to 60-80 mL in K-D evaporator. | |
| Wash with base | Transfer concentrated extract to a 1000-mL separatory funnel. Add 200 mL of petroleum ether. Extract 3X with 400 mL of 0.1 N NaOH in 10% NaCl solution. Extract 2X with 200 mL of 10% NaCl solution. | | |

TABLE 6. BATTELLE-COLUMBUS LABORATORIES PROCEDURE FOR SEMIVOLATILE ORGANICS (CONTINUED)

| Analysis scheme | Procedure | Modification | Problem/comments |
|---|---|---|---|
| A. Neutral fraction (continued) | | | |
| Dry extract | Shake final organic layer with 2 g of $MgSO_4$. | Drain pooled extracts through a column of anhydrous $Na_2SO_4$ (60 mm x 17 mm) into a K-D evaporator. | |
| Concentrate extract | Decant extract into 500-mL round-bottom flask. Concentrate to 50 mL on rotating evaporator at 35 °C. Transfer to 100-mL round-bottom flask. Concentrate to 8-10 mL. Transfer to 15-mL Vortex evaporator tube and dilute up to 10 mL with $CH_2Cl_2$. Concentrate on Vortex evaporator at 25 °C to final concentration of 200 mg of material in sample per mL of solvent. Centrifuge to remove traces of particulate matter. | Concentrate to 5 mL. Transfer to GPC cleanup column. | Reduces the number of transfers and minimizes sample losses. |

TABLE 6. BATTELLE-COLUMBUS LABORATORIES PROCEDURE FOR SEMIVOLATILE ORGANICS (CONTINUED)

| Analysis scheme | Procedure | Modifications | Problems/comments |
|---|---|---|---|
| A. Neutral fraction (continued) | | | |
| Cleanup | A 5-mL aliquot of concentrated extract is put through GPC cleanup (GPC Autoprep 1001 with a 1200 mm x 25 mm ID glass column containing Bio-Beads S-X8). Elute with $CH_2Cl_2$ at 2 mL/min for 3 h. Discard first fraction with retention time represented by minimum between di-n-tridecyl phthalate and di-n-octyl phthalate peaks. Collect Fraction GPC-1 up to retention time represented by minimum between dimethyl phthalate and 4-chlorophenyl phenyl ether peaks. Collect Fraction GPC-2 up to retention time represented by minimum between pyrene and sulfur peaks. | | If the total volume of the concentrated extract is greater than 5 mL, a second aliquot is also put through the GPC cleanup step. (Do not overload GPC column.) |
| Concentrate fraction GPC-1 | Decant extract into 500-mL round-bottom flask. Concentrate to 50 mL on rotating evaporator at 35 °C. Transfer to 100 mL round-bottom flask. Concentrate to 8-10 mL.<br>Transfer to 15-mL Vortex evaporator tube and concentrate to 2 mL. Add 2 mL of petroleum ether. | | |

TABLE 6. BATTELLE-COLUMBUS LABORATORIES PROCEDURE FOR SEMIVOLATILE ORGANICS (CONTINUED)

| Analysis scheme | Procedure | Modifications | Problems/comments |
|---|---|---|---|
| A. Neutral fraction (continued) | | | |
| Fraction GPC-1 cleanup | Apply concentrated Fraction GPC-1 to a Silica gel column (400 mm x 9 mm). Elute with 50 mL of 50% $CH_2Cl_2$ in petroleum ether and discard. Elute with 50 mL of 25% acetone in $CH_2Cl_2$ and collect as phthalate fraction. | | |
| Combine and concentrate GPC-1 and GPC-2 | Combine the phthalate fraction with Fraction GPC-2. Decant extract into 500-mL round-bottom flask. Concentrate to 50 mL on rotating evaporator at 35 °C. Transfer to 100-mL round-bottom flask. Concentrate to 10 mL. Transfer to 15-mL Vortex evaporator tube. Add 0.1 mL of anthracene-$d_{10}$ internal standard and 1.0 mL of methylene chloride. Concentrate to 0.2 mL using Vortex evaporator. | | |
| Analysis | GC/MS. | | |

TABLE 6. BATTELLE-COLUMBUS LABORATORIES PROCEDURE FOR SEMIVOLATILE ORGANICS (CONTINUED)

| Analysis scheme | Procedure | Modifications | Problems/comments |
|---|---|---|---|
| B. Acid fraction | | | |
| Sludge | Homogenize sludge. Transfer 100 g into 200-mL centrifuge tube. | Transfer 100-mL aliquot into 250-mL centrifuge tube. | |
| Acidify | Acidify with 5 g $KHSO_4$. | Homogenize for 30 sec. | Assures uniform mixing of $KHSO_4$. |
| Extract 3X | Add 100 mL of $CH_2Cl_2$. Homogenize for 1 min. Centrifuge. Remove extract with a 50-mL syringe and transfer to 500-mL round-bottom flask. Extract sample two more times. Combine extract. | Remove extract with a 100-mL glass syringe and transfer to a K-D evaporator. | |
| Dry extract | Shake combined extract with 2 g of $MgSO_4$. | Drain combined extracts through a column of anhydrous $Na_2SO_4$ (60 mm x 17 mm) into a K-D evaporator. | |

TABLE 6. BATTELLE-COLUMBUS LABORATORIES PROCEDURE FOR SEMIVOLATILE ORGANICS (CONTINUED)

| Analysis scheme | Procedure | Modifications | Problems/comments |
|---|---|---|---|
| B. Acid fraction (continued) | | | |
| Concentrate acid extract | Decant extract into 500-mL round-bottom flask. Concentrate to 50 mL on rotating evaporator at 35 °C. Transfer to 100-mL round-bottom flask. Concentrate to 8-10 mL. Transfer to 15-mL Vortex evaporator tube and dilute to 10 mL with $CH_2Cl_2$. Concentrate on Vortex evaporator at 25 °C to final concentration of 200 mg of material in sample per mL of solvent. Centrifuge to remove traces of particulate matter. | Concentrate to 8 mL and immediately transfer entire amount on GPC cleanup column. | Reduces the number of transfers and minimizes sample losses. |

TABLE 6. BATTELLE-COLUMBUS LABORATORIES PROCEDURE FOR SEMIVOLATILE ORGANICS (CONTINUED)

| Analysis scheme | Procedure | Modifications | Problems/comments |
|---|---|---|---|
| B. Acid fraction (continued) | | | |
| Cleanup | A 5-mL aliquot of concentrated extract is put through GPC cleanup (GPC Autoprep 1001 with a 1200 mm x 25 mm ID glass column) containing Bio-Beads S-X8. Elute with $CH_2Cl_2$ at 2 mL/min for 3 h. Discard first fraction with retention time represented by minimum between 4-phenylbutyric acid and 2,4-dinitrophenol peaks. Collect phenolic fraction up to retention time represented by minimum between 2,4-dichlorophenol and sulfur peaks. If total volume of the concentrated extract is greater than 5 mL, a second aliquot is also put through the cleanup step. The phenolic fractions are combined. | Transfer total concentrate or an aliquot quantitatively to GPC column (∿450 mm x 19 mm column containing Bio-Beads S-X8). Elute with $CH_2Cl_2$. Discard first fraction (∿70 mL) and collect second fraction (∿70 mL). | |

TABLE 6. BATTELLE-COLUMBUS LABORATORIES PROCEDURE FOR SEMIVOLATILE ORGANICS (CONTINUED)

| Analysis scheme | Procedure | Modifications | Problems/comments |
|---|---|---|---|
| B. Acid fraction (continued) | | | |
| Concentrate cleaned extract | Decant extract into 500-mL round-bottom flask. Concentrate to 50 mL on rotating evaporator at 35 °C. Transfer to 100-mL round-bottom flask. Concentrate to 8-10 mL. Transfer to 15-mL Vortex evaporator tube and concentrate to 2 mL. | As extract elutes from S-X8 column, pass eluent through anhydrous $Na_2SO_4$ column (60 mm x 17 mm) into K-D evaporator. Concentrate extract to ∿2 mL with K-D evaporator. | Reduces the number of transfers and minimizes sample losses. |
| Extract with base | Concentrate is transferred to 50 mL centrifuge tube. Add 200 mL of hexane. Extract 2X with 20 mL of 0.1 N NaOH in 10% NaCl solution. | Transfer concentrate to 250-mL centrifuge tube. Add 20 mL of hexane. Extract with 50 mL of 0.1 N NaOH in 10% NaCl solution. Centrifuge at 1400 R.C.F. for 5 min. | |
| Acidify | Combine aqueous layers in 200-mL centrifuge tube. Acidify with 1 mL of 6 N HCl. | Remove top layer via water aspirator. | |
| Extract 2X | Extract 2X with 20 mL $CH_2Cl_2$. Combine organic layers. | Extract 2X with 40 mL $CH_2Cl_2$. | |

TABLE 6. BATTELLE-COLUMBUS LABORATORIES PROCEDURE FOR SEMIVOLATILE ORGANICS (CONTINUED)

| Analysis scheme | Procedure | Modifications | Problems/comments |
|---|---|---|---|
| B. Acid fraction (continued) | | | |
| Dry extract | Dry over $MgSO_4$. | Drain combined extracts through a column of anhydrous $Na_2SO_4$ (60 mm x 17 mm) into a K-D evaporator. | |
| Concentrate extract | Decant extract into 500-mL round-bottom flask. Add 0.1 mL anthracene-$d_{10}$ internal standard and 1.0 mL ethylene dichloride. Concentrate to 50 mL on rotating evaporator at 35 °C. Transfer to 100-mL round-bottom flask. Concentrate to 8-10 mL. Transfer to 15-mL Vortex evaporator tube and dilute. Vortex to 10 mL with $CH_2Cl_2$. Concentrate on Vortex evaporator at 25 °C to a final concentration of 200 mg/mL of material in sample per mL of solvent. Centrifuge to remove traces of particulate matter. | | |
| Analysis | Derivation with $CH_2N_2$. Analyze by GC/MS. | | |

TABLE 6. BATTELLE-COLUMBUS LABORATORIES PROCEDURE FOR SEMIVOLATILE ORGANICS (CONTINUED)

| Analysis scheme | Procedure | Modifications | Problems/comments |
|---|---|---|---|
| C. Base fraction | | | |
| Sludge | Homogenize sludge. Transfer 10 g into 50-mL centrifuge tube. | Pour 10-mL aliquot into 250-mL centrifuge tube (round bottom, screw capped). | |
| Dilute, pH 7 buffer | Dilute with 20 mL of 0.1 M phosphate buffer, pH 7. | Dilute with 25 mL of 0.1 M phosphate buffer, pH 7. | |
| Extract 3X | Extract with 10 mL of $CHCl_3$. Homogenize for 1 min. Centrifuge. Remove $CHCl_3$ with a 50-mL syringe. Repeat extraction two more times. Combine $CHCl_3$ extracts. | Extract with 25 mL $CHCl_3$. Shake for 1 min. Centrifuge at 1400 RCF for 15 min. Remove $CHCl_3$ with 100-mL glass syringe and transfer to a 250-mL centrifuge tube. | |
| Extract with acid | Transfer $CHCl_3$ extract to 50-mL centrifuge tube. Extract 2X with 10 mL 2 N $H_2SO_4$. Remove aqueous layer with a 50-mL syringe. Combine aqueous layer in 50-mL beaker. | | |

TABLE 6. BATTELLE-COLUMBUS LABORATORIES PROCEDURE FOR SEMIVOLATILE ORGANICS (CONTINUED)

| Analysis scheme | Procedure | Modifications | Problems/comments |
|---|---|---|---|
| c. Base fraction (continued) | | | |
| Neutralize extract | Add 1 mL of 0.4 M $Na_3PO_4$. Add dropwise over 2-min period. 20% NaOH to pH 6-7. (Do not exceed pH 8.) | | |
| Extract 2X | Transfer to 60-mL separatory funnel and extract 2X with 10 mL portions of $CHCl_3$. Wash combined $CHCl_3$ extracts with 5 mL of water. | | |
| Concentrate extract | Add 5 mL of methanol to $CHCl_3$ extract. Concentrate to 0.2 mL with Vortex evaporator at 25 °C. Dilute to 1 mL with 0.1 M acetate buffer, pH 4.7. | | |
| Analysis | HPLC/EC. | | |

TABLE 7. UNIVERSITY OF WASHINGTON PROCEDURE FOR SEMIVOLATILE ORGANICS

| Analysis scheme | Procedure | Modifications | Problems/comments |
|---|---|---|---|
| Sludge | 1000 mL of mixed sample. | | |
| Acidify | Adjust to pH 2 with hydrochloric acid solution. | Recommend a smaller sample size. | Sample size too large for most solid waste. |
| Continuous extract | Liquid-liquid continuous extraction with $CH_2Cl_2$ for 12 h. Remove solvent from side vessel of continuous extractor. | | Requires large laboratory space and equipment for multiple samples to be processed at one time. |
| Basify | Adjust aqueous phase to pH 12. | | |
| Continuous extract | Liquid-liquid continuous extraction with $CH_2Cl_2$ for 12 h. Remove solvent from side vessel of continuous extractor. | | |
| Dry extracts | Dry acid and base extracts by passage through anhydrous $Na_2SO_4$. | | |
| Concentrate extracts | Concentrate acid and base extracts to 1-2 mL in K-D evaporator. | Concentrate to 10 mL or until active distillation ceases. | Cannot concentrate this volume with most waste samples |

TABLE 7. UNIVERSITY OF WASHINGTON PROCEDURE FOR SEMIVOLATILE ORGANICS (CONTINUED)

| Analysis scheme | Procedure | Modifications | Problems/comments |
|---|---|---|---|
| Cleanup | Base extract and/or acid extract are put through EPC cleanup. | Aliquots of the base and/or acid extract are put through GPC. | |
| Analysis | Because of the complexity of this procedure, details are shown in Figure 4. | | |

TABLE 8. MIDWEST RESEARCH INSTITUTE PROCEDURE FOR METALS

| Analysis scheme | Procedure | Modification | Problems/comments |
|---|---|---|---|
| A. Beryllium, Cadmium, Chromium, Copper, Nickel, Silver, Thallium, and Zinc | | | |
| 1. Preservation | | 15 mL ULTREX $HNO_3$ and 485 mL D/D $H_2O$ added to 500 mL sludge. | 1:1 sludge dilution needed to ease sample handling problems.<br>pH taken to <2. |
| 2. Digestion | Take 25 mL sludge, add 20 mL concentrated $HN_3$ and 5 mL concentrated $H_2SO_4$ and reflux. Add 10 mL concentrated $HNO_3$ and 10 mL $HClO_4$, heat until fume stage of $HClO_4$. Transfer to small beaker. Heat until volume ∿0.5 mL. | | First reflux = 4 h.<br>Second reflux = 4 h. |
| 3. Solids removal | If precipitate is formed, rinse and centrifuge three times with 5 mL 5% (v/v) $HNO_3$. Evaporate rinses to ∿15 mL. | | |
| 4. Dilution | Bring to 25 mL with 5% (v/v) $HNO_3$. | | Solutions colorless.<br>Dilution factor 2X. |
| 5. Analysis | Flame and flameless AA. | | |

TABLE 8. MIDWEST RESEARCH INSTITUTE PROCEDURE FOR METALS (CONTINUED)

| Analysis scheme | Procedure | Modifications | Problems/comments |
|---|---|---|---|
| B. Antimony, Arsenic, and Selenium | | | |
| 1. Preservation | | 15 mL ULTREX $HNO_3$ and 485 mL D/D $H_2O$ added to 500 mL sludge. | 1:1 sludge dilution needed to ease sample handling problems. pH taken to <2. |
| 2. Digestion | Take 100 mL sludge, add 20 mL concentrated $HNO_3$ and 5 mL concentrated $H_2SO_4$, and reflux 8 h. Cool, add 25 mL 30% $H_2O_2$ in 1-mL aliquots. Evaporate to less than 50 mL. Cool, add 40 mL of $HNO_3$: HCl (3/7, v/v) and reflux 2 h. | Use 50 mL sludge, add 10 mL ULTREX $HNO_3$ and 2.5 mL ULTREX $H_2SO_4$, reflux 8 h. Add 13 mL 30% $H_2O_2$. Evaporate to ∿15 mL. Add 6 mL $HNO_3$ and 14 mL ULTREX HCl. Reflux 3 h. | |
| 3. Solids removal | None. | | Black residue remaining after digestion. |
| 4. Dilution | Bring to 50 mL final volume with D/D $H_2O$. | | Solutions greenish-yellow in color. No dilution factor. |
| 5. Analysis | Hydride generation. | | |

TABLE 8. MIDWEST RESEARCH INSTITUTE PROCEDURE FOR METALS (CONTINUED)

| Analysis scheme | Procedure | Modifications | Problems/comments |
|---|---|---|---|
| C. Lead | | | |
| 1. Preservation | | 15 mL ULTREX $HNO_3$ and 485 mL D/D $H_2O$ added to 500 mL sludge. | 1:1 sludge dilution needed to ease sample handling problems.<br>pH taken to <2. |
| 2. Digestion | Take 10 mL sludge, dry at 90 °C and then dry ash for 8 h at 450 °C. Add 10 mL of 50% (v/v) $HNO_3$ and reflux 2 h. Cool, add 5 mL of $HNO_3$, and evaporate to 1 mL. | | |
| 3. Solids removal | Centrifuge and rinse three times with 5 mL 5% (v/v) $HNO_3$. Combine rinses and evaporate to less than 5 mL. | | |
| 4. Dilution | Bring to 10 mL final volume with 5% (v/v) $HNO_3$. | | Solutions golden in color. Dilution factor 2X. |
| 5. Analysis | Flame or flameless AA. | Flameless AA. | |

Table 8. MIDWEST RESEARCH INSTITUTE PROCEDURE FOR METALS (CONTINUED)

| Analysis scheme | Procedure | Modifications | Problems/comments |
|---|---|---|---|
| D. Mercury | | | |
| 1. Preservation | | 15 mL ULTREX $HNO_3$ and 485 mL D/D $H_2O$ added to 500 mL sludge. | 1:1 sludge dilution needed to ease sample handling problems.<br>pH taken to <2. |
| 2. Sample separation | 5 mL of sludge is centrifuged and supernatant decanted. Solid and supernatant treated as separate samples. | Take 10 mL of sludge. | |
| 3. Digestion supernatant | Add 1 mL concentrated $HNO_3$, 4 mL concentrated $H_2SO_4$, and reflux for 16 h. Then add 5 mL of 6% (w/v) $KMnO_4$ heated to 55 to 60 °C. React 4 h. | Used 5% $KMnO_4$. | |
| 4. Solid | Add 5 mL concentrated $HNO_3$ and 20 mL concentrated $H_2SO_4$ and reflux 80 h. Then add 25 mL 6% (w/v) $KMnO_4$ and heat to 55 to 60 °C. React 4 h. | Refluxed 16 h.<br>Used 5% $KMnO_4$. | All solids were dissolved after 16 h-reflux. |

TABLE 8. MIDWEST RESEARCH INSTITUTE PROCEDURE FOR METALS (CONTINUED)

| Analysis scheme | Procedure | Modifications | Problems/comments |
|---|---|---|---|
| D. Mercury (Continued) | | | |
| 5. Solids removal | None. | | |
| 6. Dilution | None. | Brought samples to 100 mL final volume with D/D $H_2O$. | Dilution factor 20X each fraction. |
| 7. Analysis | Cold vapor technique. | | |

## TABLE 9. BATTELLE-COLUMBUS LABORATORIES PROCEDURE FOR METALS

| Analysis scheme | Procedure | Modifications | Problems/comments |
|---|---|---|---|
| A. Flame analyses | | | |
| 1. Preservation | | 15 mL ULTREX $HNO_3$ and 485 mL D/D $H_2O$ added to 500 mL sludge. | 1:1 sludge dilution needed to ease sample handling problems. pH taken to <2. |
| 2. Digestion | Transfer 50-100 mL sludge to a 250-mL Griffin beaker, add 3 mL concentrated $HNO_3$ and evaporate to near dryness, add 3 mL concentrated $HNO_3$ and reflux with acid additions as necessary until digestion is complete. Evaporate to near dryness, then add 2 mL $HNO_3$ (1:1) per 100 mL final volume and warm solution. | Transfer 100 mL sludge to a 250 mL Griffin beaker. Add 3 mL ULTREX $HNO_3$ and evaporate to ∿15 mL. Add 3 mL $HNO_3$, reflux 2 h, add 1 mL $HNO_3$, reflux 2 h, add 2 mL $HNO_3$, and reflux 2 h. Evaporate to ∿15 mL, then add 0.5 mL ULTREX HCl and warm 15 min. | Procedure used for samples with <5% solids. |
| 3. Solids removal | Filter. | Filter through No. 42 Whatman filter. | |
| 4. Dilution | Adjust final volume based on expected metal concentrations. | Brought to final volume of 50 mL with D/D $H_2O$. | Solution golden in color. No dilution factor. |
| 5. Analysis | ICAP. | Flame AA. | |

TABLE 9. BATTELLE-COLUMBUS LABORATORIES PROCEDURE FOR METALS (CONTINUED)

| Analysis scheme | Procedure | Modifications | Problems/comments |
|---|---|---|---|
| B. Flameless analyses | | | |
| 1. Preservation | | 15 mL ULTREX $HNO_3$ and 485 mL D/D $H_2O$ added to 500 mL sludge. | 1:1 sludge dilution needed to ease sample handling problems. pH taken to <2. |
| 2. Digestion | Transfer 100 mL sludge to a 250-mL Griffin beaker. Add 3 mL concentrated $HNO_3$ and 5 mL 30% $H_2O_2$, then heat 1 h at 95 °C or until volume is reduced to less than 50 mL. | Heated until volume was ∿20 mL. | Procedure used for samples with <5% solids. |
| 3. Solids removal | None described. | Filtered through No. 42 Whatman filter. | |
| 4. Dilution | Bring to final volume of 50 mL with D/D $H_2O$. | | Solutions golden in color. No dilution factor. |
| 5. Analysis | Flameless AA. | | |

TABLE 10. UNIVERSITY OF WASHINGTON PROCEDURE FOR METALS

| Analysis scheme | Procedure | Modifications | Problems/comments |
|---|---|---|---|
| Preservation | | 15 mL ULTREX $HNO_3$ and 485 mL D/D $H_2O$ added to 500 mL sludge. | 1:1 sludge dilution needed to ease sample handling problems.<br>pH taken to <2. |
| Sample preparation | Add 20 mL sludge to clean 25-mL vials. Add ULTREX $HNO_3$ to adjust acid concentration to 2% and flame seal. | Added 0.5 mL ULTREX $HNO_3$ to 2 mL sludge. | |
| Digestion | Heat 1 h in 125 °C oven. | | |
| Solids removal | Centrifuge or filter cooled samples. | Filter through No. 41 Whatman filter. | |
| Dilution | Supernatant or filtrate taken without volume adjustment. | | Solutions golden in color, dilution factor 2X. |
| Analysis | Analyze by ICAP; Hg by cold vapor method. | Analyze by flame and flameless AA; Hg by cold vapor method. | |

TABLE 11. EMSL-Ci INTERIM METHOD FOR METALS

| Analysis scheme | Procedure | Modifications | Problems/comments |
|---|---|---|---|
| 1. Preservation | | 15 mL ULTREX $HNO_3$ and 485 mL D/D $H_2O$ added to 500 mL sludge. | 1:1 sludge dilution needed to ease sample handling problems.<br>pH taken to <2. |
| 2. Digestion | 1 gm of sludge dried at 60 °C, pulverized, and mixed in 125-mL conical Phillips beaker. Add 5 mL $HNO_3$ (1:1) and reflux to near dryness. Cool, add 4 mL concentrated $HNO_3$ and reflux to near dryness. Cool, add 1 mL $HNO_3$ (1:1) and 3 mL 30% $H_2O_2$ additions (not to exceed 10 mL total) until effervescence is minimal. | 25 mL of sludge in a 125- Erlenmeyer flask. Add 2.5 mL ULTREX $HNO_3$ and reflux 8 h, remove watch glass and evaporate to ~15 mL. Add 0.5 mL $HNO_3$ and 3 mL 30% $H_2O_2$ and heat. Cool, add 3 mL $H_2O_2$, then heat; repeat twice. | 25 mL of 1:1 dilution sludge contains about 0.5 g solids.<br>Color removed on addition of $H_2O_2$. |
| For flame preparation | Add 2 mL HCl (1:1) and reflux 10 min. | | |
| For flameless preparation | Add 2 mL $HNO_3$ (1:1) and reflux 10 min. | | |
| 3. Solids removal | Filter through No. 42 Whatman filter. | | |
| 4. Dilution | Bring to final volume of 50 mL with D/D $H_2O$. | | Solutions essentially colorless. Dilution factor of 4. |
| 5. Analyses | Flame and flameless AA. | | |

TABLE 12. ENVIRONMENTAL PROTECTION AGENCY PROCEDURE FOR METALS

| Analysis scheme | Procedure | Modifications | Problems/comments |
|---|---|---|---|
| A. Preparation for total recoverable metals | | | |
| 1. Preservation | 5 mL $HNO_3$/L sample. | 15 mL ULTREX $HNO_3$ and 485 mL D/D $H_2O$ added to 500 mL sludge. | 1:1 sludge dilution needed to ease sample handling problems.<br>pH taken to <2. |
| 2. Digestion-flame AA | Transfer 100 mL of sludge to a beaker or flask. Add 5 mL distilled HCl (1:1). Heat, without boiling, to 15-20 mL. | | |
| 3. Digestion-flameless AA | Transfer 100 mL of sludge to a beaker or flask. Heat, without boiling, until concentrated to 15-20 mL. | Add 1 mL ULTREX $HNO_3$ and heat 2 h, add 2 mL ULTREX $HNO_3$ and continue heating. | No visible change in appearance with additional digestion. |
| 4. Solids removal | Filter. | Suction filter through No. 42 Whatman filter. | Extremely difficult to suction filter these samples through No. 42 paper. |
| 5. Dilution | Bring to 100 mL or less to effect concentration. | Brought to 50 mL final volume with D/D $H_2O$. | Solution golden to light brown. No dilution factor. |
| 6. Analysis | Flame or flameless AA. | | |

TABLE 12. ENVIRONMENTAL PROTECTION AGENCY PROCEDURE FOR METALS (CONTINUED)

| Analysis scheme | Procedure | Modifications | Problems/comments |
|---|---|---|---|
| B. Manual cold vapor technique for mercury | | | |
| 1. Preservation | 5 mL $HNO_3$/L upon collection. | 15 mL ULTREX $HNO_3$ and 485 mL D/D $H_2O$ added to 500 mL sludge. | 1:1 sludge dilution needed to ease sample handling problems.<br>pH taken to <2. |
| 2. Sample preparation | Take 100 mL sludge in 300 mL BOD bottle, add 5 mL $H_2SO_4$, 2.5 mL concentrated $HNO_3$ and mix. Add 15 mL 5% $KMnO_4$ solution (samples may require more $KMnO_4$), 8 mL 5% potassium persulfate solution, and heat 2 h at 95 °C. Cool, add 6 mL 12% sodium chloride—hydroxylamine sulfate solution. | Use 2 mL of sludge. | |
| 3. Analysis | After >30 sec delay, add 5 mL 10% stannous sulfate solution. Immediately attach the bottle to the aeration apparatus and sparge. | | |

The MRI and EPA methods use a commercial purge-and-trap (PAT) apparatus such as the Tekmar Liquid Sample Concentrator Model LSC-1 or its equivalent coupled to the GC/MS instrumentation. The University of Washington evaluated the use of a commercial purge-and-trap apparatus and compared the accuracy and precision with that obtained by an alternate procedure. Thus, the UW procedure offered alternatives depending on the availability of a specific purging apparatus. The alternate procedure involves the use of a PAT constructed from components in the laboratory. Generally, the method requires larger samples, longer purge times, elevated temperature of the purge chamber, and higher flow rates. The sorbent trap detaches from the system and is manually connected to the GC/MS instrumentation for desorption with an external heating system. Generally, recommended operating parameters for the alternate method were considered to be more difficult to manage in the analysis of a large number of samples; however, the apparatus does offer the advantage of being able to purge multiple samples in the field that require immediate analysis at the time of collection. Sorbent traps can be closed and later analyzed in the laboratory.

The gas chromatographic program was essentially identical in all the methods with the exception of the UW procedure, which recommended a program that exceeded the suggested temperature limit of the column packing.

All methods used chromatographic columns of either 1/8 in. OD stainless steel, or 2 mm ID glass packed with 0.2% of Carbowax 1500 on Carbopack C, 60/80 mesh. The length of the columns, however, varied from 2.4 to 3.7 m.

In reviewing the relative merits of the four procedures, there appeared to be no substantial differences among the MRI, EPA, and the very similar EPA-IM methods. The UW method was considered to be somewhat less desirable with respect to analysis time and simplicity of operation. Therefore, a combination of the MRI and EPA procedures, with minor modifications, was selected as the method of choice.

The modifications recommended by SoRI include the use of commercially available, prepacked, sorbent traps to eliminate the reproducibility problems encountered in packing sorbent columns. The purge chamber was modified by replacing the septum inlet with a ground glass joint and closure to permit the introduction of solid and viscous samples into the purge chamber. Glass, rather than stainless steel, should be used as the analytical column material since glass columns allow the analyst to note conditions such as material collecting at the injection port and on the column packing. Finally, 1% of SP 1000 on Carbopack B, 60/80 mesh should be used as the column packing. This material afforded better separation of compounds and tolerated the higher temperature programs which were often necessary to clean and condition contaminated columns.

Because of the diversity and complexity of waste samples, preliminary spiking tests should be conducted with each particular type of sample to determine the applicability of the proposed method. Further procedural modifications may be necessary to adapt the method for use with certain waste materials. A summary of the methods and modifications is given in Tables 1 through 4.

## SEMIVOLATILE ORGANICS

Three methods for determination of semivolatile organics in residual wastes were selected based on the recommendations of Effluent Guidelines Division for evaluation: Midwest Research Institute (MRI),[1] Battelle-Columbus Laboratories (BCL),[2] and the University of Washington (UW).[3] Each procedure was first reviewed critically to determine its utility according to the criteria for this study. Flow charts for the three procedures are given in Figures 1 through 3, while condensed versions of the methods, modifications, problems, and comments are shown in Tables 5 through 7. Because of the complexity of the UW procedure, Figure 4 presents the flow chart in greater detail.

A comparison of major events in the analytical schemes of each procedure, such as number of extractions, Kuderna-Danish concentrations, column chromatographic cleanups, and individual analyses are presented in Table 13. The UW procedure required 26 concentration steps and 8 to 12 GC/MS analyses of final concentrates when the acid extract was not combined with the base/neutral extract prior to the gel permeation chromatographic cleanup step. Based on the complexity of the method, the number of analyses required, and time involved in sample preparation, the UW procedure was eliminated from further consideration.

The BCL method required that three aliquots of the waste be extracted for acidic, neutral, and basic compounds, respectively. The MRI method, however, required the extraction of only a single aliquot of sample to obtain the same acidic, neutral, and basic components. Therefore, the MRI procedure was selected for an extended evaluation with Coke Oven Biological Sludge, Food Processing Waste, and Lime Slurry Treatment Sludge.

It should be noted that a revised BCL procedure, requiring the extraction of only one aliquot of waste material, was received for consideration, but only after the MRI evaluation was completed.

The MRI procedure, with the minor modifications recommended by SoRI, is shown in Table 5. These modifications include (1) the extraction of only one aliquot when the suspended solids content of the sample is low, (2) the alternative use of descending liquid column chromatography techniques instead of the gel permeation chromatography with an Autoprep 1002, and (3) the use of more concentrated acidic and basic solutions to adjust the sample pH, thereby avoiding excessive aqueous dilution of the sample.

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1. "Development of Analytical Test Procedure for the Measurement of Organic Priority Pollutants in Sludge and Sediments," Midwest Research Institute Final Report EPA Contract No. 68-03-2695, June 26, 1979.

2. "Development of Analytical Protocols for Organic Priority Pollutants in Municipal Sludges," Battelle-Columbus Laboratories, Final Report EPA Contract No. 68-03-2624, March 30, 1979.

3. "Presence of Priority Pollutants in Sewage and their Removal on Sewage Treatment Plants," University of Washington, Annual Report Grant R.806102, July 31, 1979.

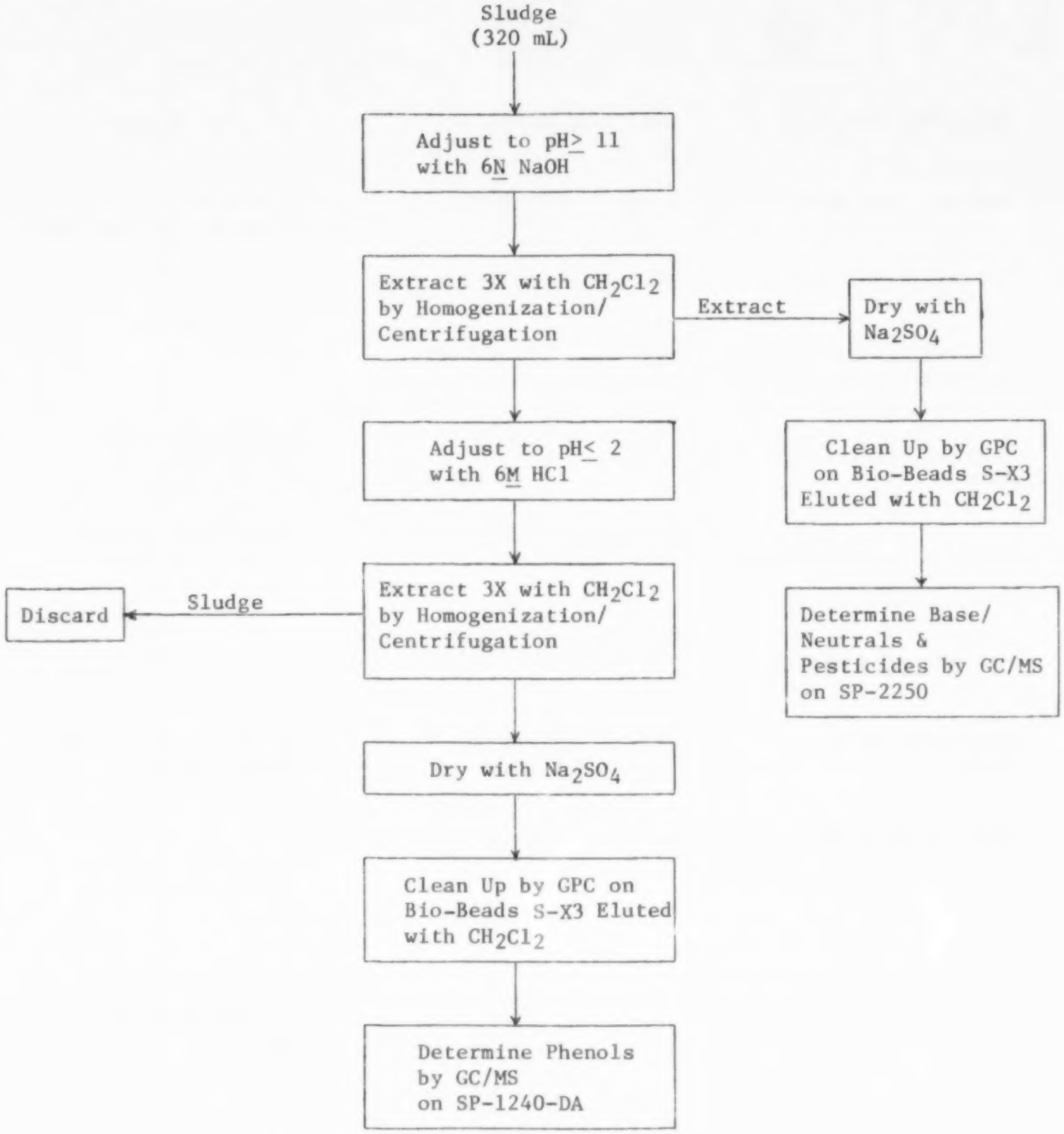

Sludge
(320 mL)
Adjust to pH$\geq$ 11 with 6N NaOH
Extract 3X with $CH_2Cl_2$ by Homogenization/ Centrifugation
Extract
Dry with $Na_2SO_4$
Adjust to pH$\leq$ 2 with 6M HCl
Clean Up by GPC on Bio-Beads S-X3 Eluted with $CH_2Cl_2$
Discard
Sludge
Extract 3X with $CH_2Cl_2$ by Homogenization/ Centrifugation
Determine Base/ Neutrals & Pesticides by GC/MS on SP-2250
Dry with $Na_2SO_4$
Clean Up by GPC on Bio-Beads S-X3 Eluted with $CH_2Cl_2$
Determine Phenols by GC/MS on SP-1240-DA

Figure 1. Midwest Research Institute procedure for semivolatile organics.

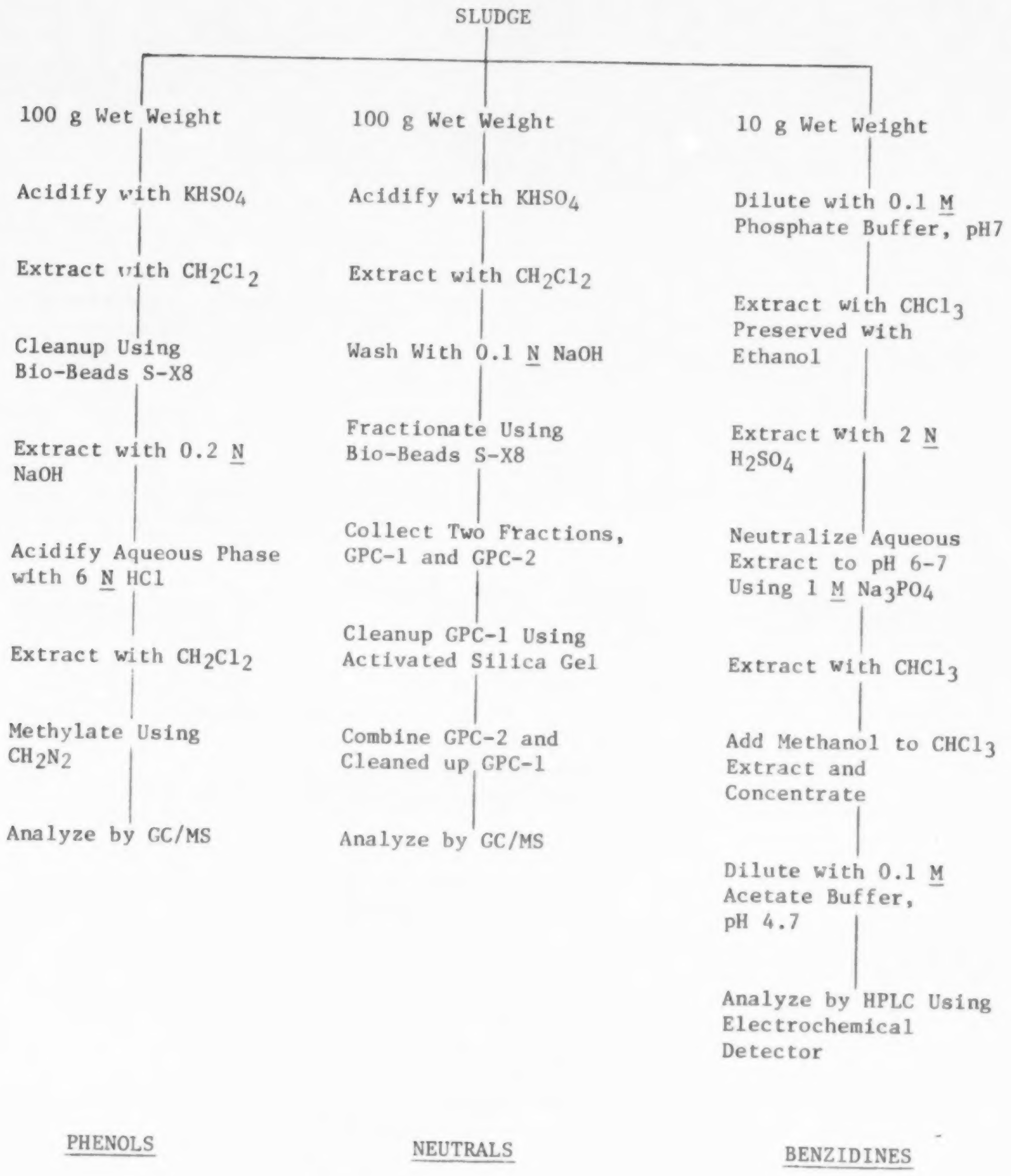
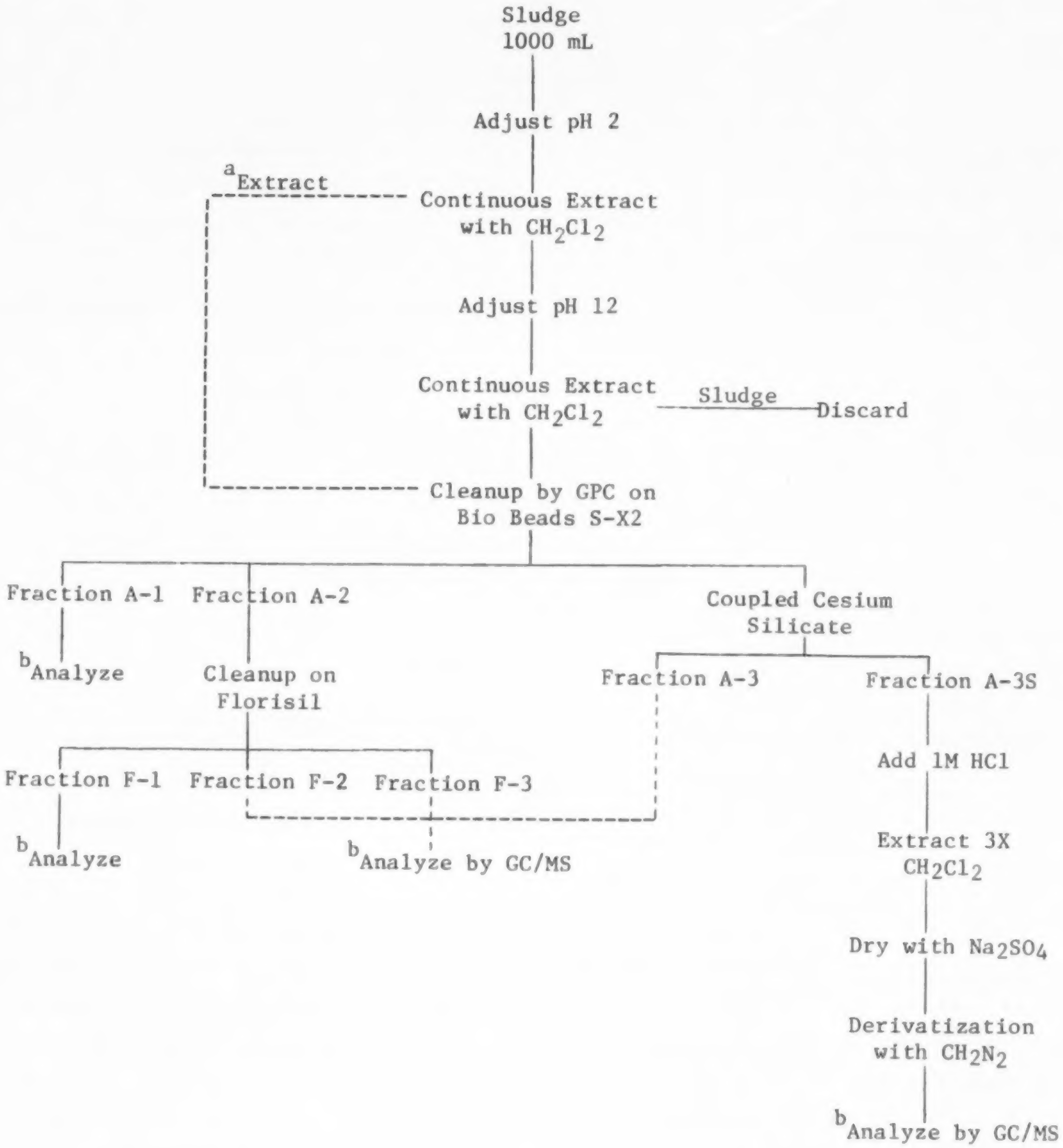

SLUDGE
100 g Wet Weight
Acidify with $KHSO_4$
Extract with $CH_2Cl_2$
Cleanup Using Bio-Beads S-X8
Extract with 0.2 N NaOH
Acidify Aqueous Phase with 6 N HCl
Extract with $CH_2Cl_2$
Methylate Using $CH_2N_2$
Analyze by GC/MS
100 g Wet Weight
Acidify with $KHSO_4$
Extract with $CH_2Cl_2$
Wash With 0.1 N NaOH
Fractionate Using Bio-Beads S-X8
Collect Two Fractions, GPC-1 and GPC-2
Cleanup GPC-1 Using Activated Silica Gel
Combine GPC-2 and Cleaned up GPC-1
Analyze by GC/MS
10 g Wet Weight
Dilute with 0.1 M Phosphate Buffer, pH7
Extract with $CHCl_3$ Preserved with Ethanol
Extract with 2 N $H_2SO_4$
Neutralize Aqueous Extract to pH 6-7 Using 1 M $Na_3PO_4$
Extract with $CHCl_3$
Add Methanol to $CHCl_3$ Extract and Concentrate
Dilute with 0.1 M Acetate Buffer, pH 4.7
Analyze by HPLC Using Electrochemical Detector
PHENOLS
NEUTRALS
BENZIDINES

Figure 2. Battelle-Columbus Laboratories procedure for semivolatile organics.


Sludge
1000 mL
Adjust pH 2
[a]Extract
Continuous Extract with $CH_2Cl_2$
Adjust pH 12
Continuous Extract with $CH_2Cl_2$
Sludge
Discard
Cleanup by GPC on Bio Beads S-X2
Fraction A-1
Fraction A-2
Coupled Cesium Silicate
[b]Analyze
Cleanup on Florisil
Fraction A-3
Fraction A-3S
Fraction F-1
Fraction F-2
Fraction F-3
Add 1M HCl
Extract 3X $CH_2Cl_2$
[b]Analyze
[b]Analyze by GC/MS
Dry with $Na_2SO_4$
Derivatization with $CH_2N_2$
[b]Analyze by GC/MS

a. Base extract separate from or combined with acid extract.

b. Capillary GC-FID screening.
Capillary GC/MS analysis.
GC/EC analysis.

Figure 3. Summary of University of Washington procedure for semivolatile organics.

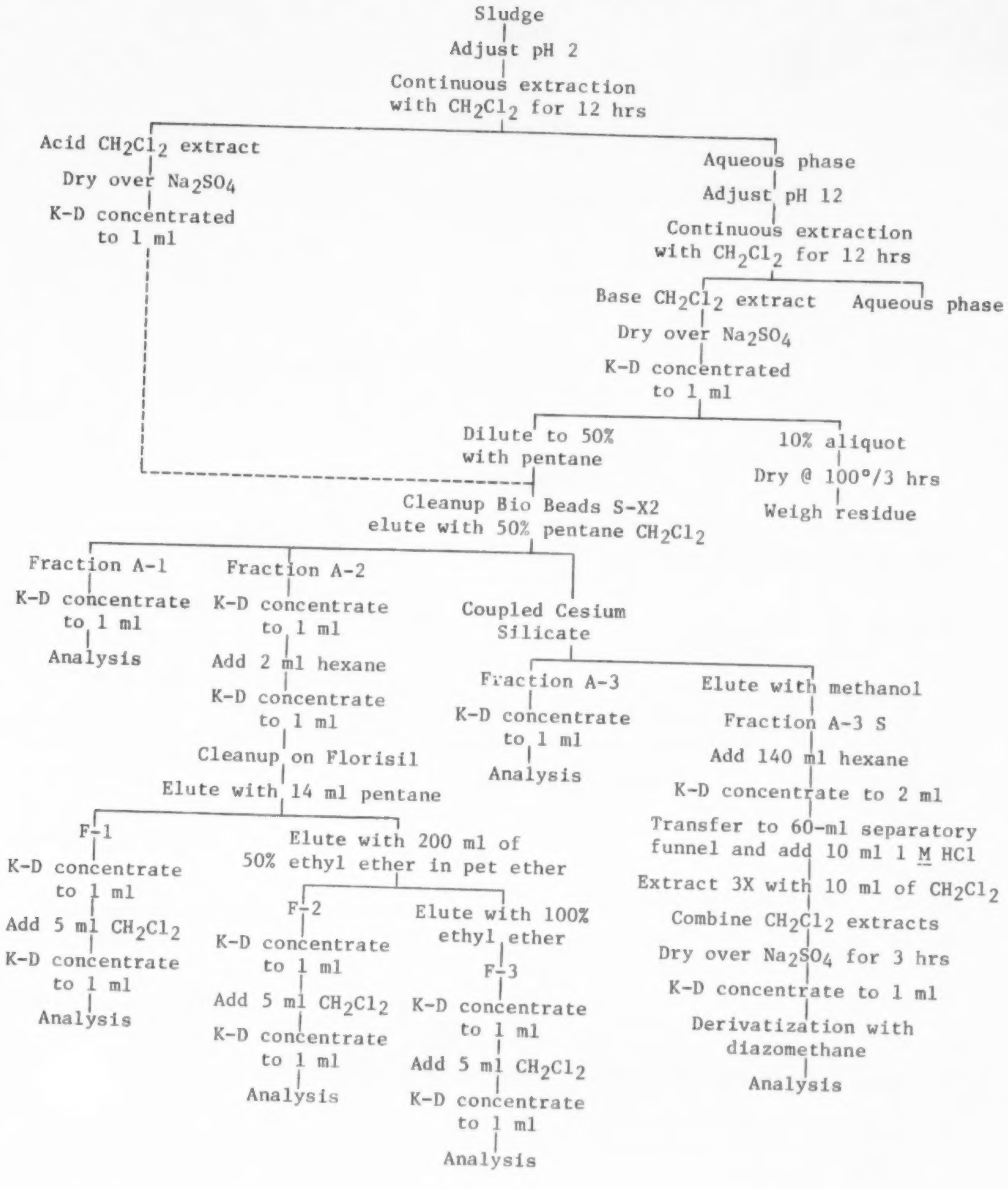

Sludge
Adjust pH 2
Continuous extraction with $CH_2Cl_2$ for 12 hrs
Acid $CH_2Cl_2$ extract
Dry over $Na_2SO_4$
K-D concentrated to 1 ml
Aqueous phase
Adjust pH 12
Continuous extraction with $CH_2Cl_2$ for 12 hrs
Base $CH_2Cl_2$ extract
Aqueous phase
Dry over $Na_2SO_4$
K-D concentrated to 1 ml
Dilute to 50% with pentane
10% aliquot
Dry @ 100°/3 hrs
Weigh residue
Cleanup Bio Beads S-X2 elute with 50% pentane $CH_2Cl_2$
Fraction A-1
K-D concentrate to 1 ml
Analysis
Fraction A-2
K-D concentrate to 1 ml
Add 2 ml hexane
K-D concentrate to 1 ml
Cleanup on Florisil
Elute with 14 ml pentane
F-1
K-D concentrate to 1 ml
Add 5 ml $CH_2Cl_2$
K-D concentrate to 1 ml
Analysis
Elute with 200 ml of 50% ethyl ether in pet ether
F-2
K-D concentrate to 1 ml
Add 5 ml $CH_2Cl_2$
K-D concentrate to 1 ml
Analysis
Elute with 100% ethyl ether
F-3
K-D concentrate to 1 ml
Add 5 ml $CH_2Cl_2$
K-D concentrate to 1 ml
Analysis
Coupled Cesium Silicate
Fraction A-3
K-D concentrate to 1 ml
Analysis
Elute with methanol
Fraction A-3 S
Add 140 ml hexane
K-D concentrate to 2 ml
Transfer to 60-ml separatory funnel and add 10 ml 1 M HCl
Extract 3X with 10 ml of $CH_2Cl_2$
Combine $CH_2Cl_2$ extracts
Dry over $Na_2SO_4$ for 3 hrs
K-D concentrate to 1 ml
Derivatization with diazomethane
Analysis

Figure 4. Detailed analyses of University of Washington procedure for semivolatile organics.

TABLE 13. COMPARISON OF RELATIVE MAJOR EVENTS IN THE ANALYTICAL SCHEMES FOR SEMIVOLATILE ORGANICS

| Event | Midwest Research Institute | Battelle-Columbus Laboratories | University of Washington |
|---|---|---|---|
| Extractions | 6 | 22 | 8 |
| K-D concentrations | 6 | 20 | 26 |
| Cleanup | 2 | 3 | 6 |
| Analysis | 2 | 3 | 12 |
| Total | 16 | 48 | 52 |

The MRI procedure was evaluated further with samples of waste from Coke Oven Biological Treatment, Food Processing, and Lime Treatment operations. Samples and spiked samples were analyzed in duplicate, and recovery data were calculated for selected semivolatile organics. The results of these analyses are presented in Tables 14 through 21.

The results indicated that recovery varied with the type of waste. For the most part, good recoveries were obtained for the Food Processing Sludge and the Lime Treatment Sludge. Recoveries of selected compounds from the Coke Oven Biological Sludge were generally low, varying from near zero to 75%. Although low recoveries from the Biological Sludge were probably due to matrix effect involving adsorption on suspended particulates, they can also be attributed in part to physical losses occurring in the extraction steps of the procedure.

Based on these preliminary surveys of existing methods, all of which used extraction, concentration, and analysis by GC/MS, the modified MRI method was selected for further evaluation. Furthermore, the preliminary analyses for organics in the three wastes produced results that were supportive. Therefore, the EPA-MRI procedure for purgeable organic compounds and the modified MRI procedure for the analysis of semivolatile organic compounds were selected for validation with other residual wastes. Results of this in-depth study are presented in Section 5.

## METALS

### Comparison of Digestion Procedures with Coke Oven Sludge

Based on recommendations from EGD, four digestion procedures developed by Midwest Research Institute (MRI),[1] Environmental Monitoring and Support Laboratories of Cincinnati (EMSL-Ci),[2] Battelle-Columbus Laboratories (BCL),[3] and the University of Washington (UW)[4] were evaluated initially. A Coke Oven Sludge was used to compare the relative extraction efficiencies of the procedures for the selected metals. The EPA procedure for Total Recoverable Metals[5] was also included for comparison since this procedure was used to

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[1] "Effluent Guidelines Division POTW Sampling and Analysis," Midwest Research Institute, Progress Report on EPA Contract No. 68-03-2565, November 14, 1979.

[2] "Interim Method for the Analysis of Elemental Priority Pollutants in Sludge," Environmental Monitoring and Support Laboratories in Cincinnati, December 1978.

[3] "ICAP Digestion Procedures for Metals - Aqueous Solutions," Battelle-Columbus Laboratories, December 3, 1979.

[4] "Procedure of Analysis of Heavy Metals and Other Elements in Sewage or Sludge," University of Washington, Laboratory of Radiation Ecology Research Report, February 21, 1980.

[5] "Methods for Chemical Analysis of Water and Waste," EPA, March 1979.

## TABLE 14. GC/MS DATA—PERCENT RECOVERY OF SEMIVOLATILE BASE/NEUTRAL EXTRACTABLE ORGANICS IN DISTILLED WATER

| Compound | Retention time, min | Ion used for quantitation | Conc found in unspiked samples, μg/L No.1 | Conc found in unspiked samples, μg/L No.2 | Conc found in spiked samples, μg/L No.1 | Conc found in spiked samples, μg/L No.2 | Average recovery, % |
|---|---|---|---|---|---|---|---|
| 1,3-Dichlorobenzene | 3.8 | 146 | - | - | 930 | 840 | 89 |
| 1,4-Dichlorobenzene | 4.2 | 146 | - | - | 913 | 1020 | 97 |
| 1,2-Dichlorobenzene | 4.9 | 146 | - | - | 992 | 1050 | 102 |
| Bis(2-chloroethyl) ether | 6.8 | 93 | - | - | 1090 | 1070 | 104 |
| Hexachloroethane | 4.8 | 117 | - | - | 926 | 905 | 92 |
| Bis(2-chloroisopropyl) ether | 6-7 | 45 | - | - | 1000 | 967 | 99 |
| Nitrobenzene | 9.0 | 77 | - | - | 1270 | 1190 | 123 |
| Hexachlorobutadiene | 8.5 | 225 | - | - | 983 | 929 | 96 |
| Naphthalene | 9.2 | 128 | - | - | 1460 | 1040 | 125 |
| 1,2,4-Trichlorobenzene | 9.6 | 180 | - | - | 1060 | 1020 | 104 |
| Bis(2-chloroethoxy) methane | 10-12 | 93 | - | - | 1080 | 929 | 101 |
| N-nitrosodipropylamine | 10.5-12.5 | 130/70 | - | - | 1010 | 953 | 98 |
| Hexachlorocyclopentadiene | 11.1 | 237 | - | - | 842 | 811 | 84 |
| 2-Chloronaphthalene | 13.0 | 162 | - | - | 1000 | 920 | 96 |
| Isophorone | 13-15 | 82 | - | - | 934 | 847 | 91 |
| Acenaphthylene | 14.3 | 152 | - | - | 935 | 882 | 91 |
| Dimethyl phthalate | 16-17 | 163 | - | - | 1380 | 1440 | 71 |
| 2,6-Dinitrotoluene | 15.9 | 165 | - | - | 854 | 852 | 85 |
| 2,4-Dinitrotoluene | 17.1 | 165 | - | - | 847 | 855 | 85 |
| Acenaphthene | 14.9 | 154 | - | - | 885 | 859 | 87 |
| Fluorene | 16.4 | 166 | - | - | 901 | 893 | 90 |
| Diethyl phthalate | 17.6 | 149 | - | - | 1490 | 1540 | 76 |
| N-nitrosodiphenylamine | 17.6 | 169 | - | - | 1340 | 1460 | 140 |
| 1,2-Diphenylhydrazine | 17.1 | 77 | - | - | 930 | 838 | 88 |
| Hexachlorobenzene | 17.8 | 282 | - | - | 849 | 975 | 91 |
| 4-Bromophenyl phenyl ether | 18.1 | 248 | - | - | 886 | 1020 | 92 |
| Phenanthrene/anthracene | 19.7 | 178 | - | - | 2000 | 1950 | 99 |
| Di-n-butyl phthalate | 22.0 | 149 | - | - | 980 | 994 | 97 |
| Fluoranthrene | 23.4 | 202 | - | - | 987 | 971 | 98 |
| Pyrene | 24.1 | 202 | - | - | 984 | 978 | 98 |
| Benzidine | 26.0 | 184 | - | - | 825 | <100 | 41 |
| Chrysene/benzo(a)anthracene | 28.0 | 228 | - | - | 2340 | 2230 | 114 |
| Butylbenzyl phthalate | 27.0 | 149 | - | - | 986 | 902 | 94 |
| Bis(2-ethylhexyl) phthalate | 27.7 | 149 | - | - | 2110 | 1930 | 101 |
| Di-n-octyl phthalate | 29.7 | 149 | - | - | 1140 | 1080 | 111 |
| Benzo(b)fluoranthene/ benzo(k)fluoranthene | 31.2 | 252 | - | - | 2550 | 2510 | 127 |
| Benzo(a)pyrene | 32.3 | 252 | - | - | 1360 | 1380 | 137 |
| 3,3'-Dichlorobenzidine | 29.3 | 252 | - | - | 970 | 1030 | 100 |
| Benzo(ghi)perylene | 38.2 | 276 | - | - | 1660 | 1510 | 158 |
| Indeno(1,2,3-cd)pyrene | 36.7 | 276 | - | - | 1420 | 1360 | 139 |
| Dibenzo(ah)anthracene | 36.9 | 278 | - | - | 1550 | 1410 | 148 |

## TABLE 15. GC/MS DATA—PERCENT RECOVERY OF SEMIVOLATILE ACID EXTRACTABLE ORGANICS IN DISTILLED WATER

| Compound | Retention time, min | Ion used for quantitation | Conc found in unspiked samples, µg/L | | Conc found in spiked samples,* µg/L | | Average recovery, % |
|---|---|---|---|---|---|---|---|
| | | | No.1 | No.2 | No.1 | No.2 | |
| 2-Chlorophenol | 4.7 | 128 | - | - | 1140 | 1050 | 88 |
| 2-Nitrophenol | 5.1 | 139 | - | - | 1210 | 1130 | 94 |
| Phenol | 6.8 | 94 | - | - | 1050 | 868 | 77 |
| 2,4-Dimethylphenol | 8.3 | 122 | - | - | 355 | 380 | 29 |
| 2,4-Dichlorophenol | 8.7 | 162 | - | - | 1150 | 1120 | 91 |
| 2,4,6-Trichlorophenol | 10.6 | 196 | - | - | 1190 | 1220 | 96 |
| 4-Chloro-3-methylphenol | 12.2 | 142 | - | - | 1110 | 1130 | 90 |
| 2,4-Dinitrophenol | 15-16 | 184 | - | - | 1530 | 1830 | 134 |
| Pentachlorophenol | 16.4 | 266 | - | - | 1210 | 1370 | 103 |
| 4-Nitrophenol | 20.0 | 139/65 | - | - | 1340 | 1250 | 104 |

* Concentration spike—1250 µg/L.

### TABLE 16. GC/MS DATA—PERCENT RECOVERY OF SEMIVOLATILE BASE/NEUTRAL EXTRACTABLE ORGANICS IN COKE OVEN BIOLOGICAL SLUDGE

| Compound | Retention time, min | Ion used for quantitation | Conc found in unspiked samples, µg/L | | Conc found in spiked samples, µg/L | | Average recovery, % |
|---|---|---|---|---|---|---|---|
| | | | No.1 | No.2 | No.1 | No.2 | |
| 1,3-Dichlorobenzene | 3.8 | 146 | ND | ND | 331 | 444 | 39 |
| 1,4-Dichlorobenzene | 4.2 | 146 | ND | ND | 482 | 529 | 51 |
| 1,2-Dichlorobenzene | 4.9 | 146 | ND | ND | 641 | 584 | 61 |
| Bis(2-chloroethyl) ether | 6.8 | 93 | ND | ND | 715 | 848 | 75 |
| Hexachloroethane | 4.8 | 117 | ND | ND | 323 | 257 | 29 |
| Bis(2-chloroisopropyl) ether | 6-7 | 45 | ND | ND | 574 | 649 | 61 |
| Nitrobenzene | 9.0 | 77 | ND | ND | 717 | 723 | 72 |
| Hexachlorobutadiene | 8.5 | 225 | ND | ND | 252 | 179 | 22 |
| Naphthalene | 9.2 | 128 | ND | ND | 495 | 442 | 47 |
| 1,2,4-Trichlorobenzene | 9.6 | 180 | ND | ND | 429 | 374 | 40 |
| Bis(2-chloroethoxy) methane | 10-12 | 93 | <100 | <100 | 469 | 599 | 46 |
| N-nitrosodipropylamine | 10.5-12.5 | 130/70 | ND | ND | 556 | 604 | 58 |
| Hexachlorocyclopentadiene | 11.1 | 237 | ND | ND | <100 | <100 | <10 |
| 2-Chloronaphthalene | 13.0 | 162 | ND | ND | 321 | 262 | 29 |
| Isophorone | 13-15 | 82 | ND | ND | 554 | 625 | 59 |
| Acenaphthylene | 14.3 | 152 | <100 | <100 | 335 | 308 | 27 |
| Dimethyl phthalate | 16-17 | 163 | ND | ND | 644 | 827 | 37 |
| 2,6-Dinitrotoluene | 15.9 | 165 | ND | ND | 360 | 504 | 43 |
| 2,4-Dinitrotoluene | 17.1 | 165 | ND | ND | 438 | 625 | 53 |
| Acenaphthene | 14.9 | 154 | ND | ND | 266 | 260 | 26 |
| Fluorene | 16.4 | 166 | ND | ND | 282 | 224 | 25 |
| Diethyl phthalate | 17.6 | 149 | ND | ND | 714 | 684 | 35 |
| N-nitrosodiphenylamine | 17.6 | 169 | ND | ND | 582 | 476 | 53 |
| 1,2-Diphenylhydrazine | 17.1 | 77 | ND | ND | 252 | 170 | 21 |
| Hexachlorobenzene | 17.8 | 282 | ND | ND | 200 | 143 | 17 |
| 4-Bromophenyl phenyl ether | 18.1 | 248 | ND | ND | 230 | 164 | 20 |
| Phenanthrene/anthracene | 19.7 | 178 | <100 | <100 | 571 | 371 | 22 |
| Di-n-butyl phthalate | 22.0 | 149 | ND | ND | 171 | 113 | 14 |
| Fluoranthrene | 23.4 | 202 | <100 | <100 | 228 | 147 | 13 |
| Pyrene | 24.1 | 202 | 113 | 134 | 284 | 170 | 10 |
| Benzidine | 26.0 | 184 | ND | ND | 2910 | 2230 | 257 |
| Chrysene/benzo(a)anthracene | 28.0 | 228 | 524 | 627 | 695 | 361 | <10 |
| Butylbenzyl phthalate | 27.0 | 149 | <100 | ND | 136 | <100 | 10 |
| Bis(2-ethylhexyl) phthalate | 27.7 | 149 | 590 | 979 | 589 | 208 | <10 |
| Di-n-octyl phthalate | 29.7 | 149 | ND | ND | 196 | <100 | 13 |
| Benzo(b)fluoranthene/ benzo(k)fluoranthene | 31.2 | 252 | 1980 | 2290 | 1620 | 678 | <10 |
| Benzo(a)pyrene | 32.3 | 252 | 1720 | 1870 | 1430 | 548 | <10 |
| 3,3'-Dichlorobenzidine | 29.3 | 252 | ND | ND | 668 | 132 | 40 |
| Benzo(ghi)perylene | 38.2 | 276 | 1170 | NA | 1230 | 341 | <10 |
| Indeno(1,2,3-cd)pyrene | 36.7 | 276 | 1460 | NA | 1340 | 395 | <10 |
| Dibenzo(ah)anthracene | 36.9 | 278 | 468 | NA | 817 | 202 | <10 |

Note: ND = Not detected.
NA = Not analyzed.

### TABLE 17. GC/MS DATA—PERCENT RECOVERY OF SEMIVOLATILE ACID EXTRACTABLE ORGANICS IN COKE OVEN BIOLOGICAL SLUDGE

| Compound | Retention time, min | Ion used for quantitation | Conc found in unspiked samples, μg/L No.1 | Conc found in unspiked samples, μg/L No.2 | Conc found in spiked samples,* μg/L No.1 | Conc found in spiked samples,* μg/L No.2 | Average recovery, % |
|---|---|---|---|---|---|---|---|
| 2-Chlorophenol | 4.7 | 128 | ND | ND | 693 | 773 | 59 |
| 2-Nitrophenol | 5.1 | 139 | ND | ND | 587 | 754 | 54 |
| Phenol | 6.8 | 94 | 640 | 338 | 1040 | 1110 | 47 |
| 2,4-Dimethylphenol | 8.3 | 122 | <250 | <250 | 464 | 518 | 37 |
| 2,4-Dichlorophenol | 8.7 | 162 | ND | ND | 612 | 690 | 52 |
| 2,4,6-Trichlorophenol | 10.6 | 196 | ND | ND | 566 | 741 | 52 |
| 4-Chloro-3-methylphenol | 12.2 | 142 | <250 | <250 | 578 | 706 | 44 |
| 2,4-Dinitrophenol | 15-16 | 184 | ND | ND | 1050 | 784 | 73 |
| Pentachlorophenol | 16.4 | 266 | ND | ND | 542 | 568 | 44 |
| 4-Nitrophenol | 20.0 | 139/65 | ND | ND | 1140 | 1230 | 95 |

Note: ND = Not detected.

* Concentration spike—1250 μg/L.

TABLE 18. GC/MS DATA—PERCENT RECOVERY OF SEMIVOLATILE BASE/NEUTRAL EXTRACTABLE ORGANICS IN FOOD PROCESSING SLUDGE

| Compound | Retention time, min | Ion used for quantitation | Conc found in unspiked samples, µg/L | | Conc found in spiked samples, µg/L | | Average recovery, % |
|---|---|---|---|---|---|---|---|
| | | | No.1 | No.2 | No.1 | No.2 | |
| 1,3-Dichlorobenzene | 3.8 | 146 | ND | ND | 933 | 303 | 62 |
| 1,4-Dichlorobenzene | 4.2 | 146 | ND | ND | 1130 | 529 | 83 |
| 1,2-Dichlorobenzene | 4.9 | 146 | ND | ND | 1210 | 662 | 93 |
| Bis(2-chloroethyl) ether | 6-8 | 93 | ND | ND | 1220 | 667 | 91 |
| Hexachloroethane | 4.8 | 117 | ND | ND | 1020 | 504 | 76 |
| Bis(2-chloroisopropyl) ether | 6-7 | 45 | ND | ND | 1120 | 597 | 86 |
| Nitrobenzene | 9.0 | 77 | ND | ND | 1130 | 604 | 87 |
| Hexachlorobutadiene | 8.5 | 225 | ND | ND | 1080 | 546 | 81 |
| Naphthalene | 9.2 | 128 | ND | ND | 1200 | 633 | 92 |
| 1,2,4-Trichlorobenzene | 9.6 | 180 | ND | ND | 1110 | 642 | 88 |
| Bis(2-chloroethoxy) methane | 10-12 | 93 | ND | ND | 1140 | 544 | 84 |
| N-nitrosodipropylamine | 10.5-12.5 | 130/70 | ND | ND | 944 | 396 | 67 |
| Hexachlorocyclopentadiene | 11.1 | 237 | ND | ND | 678 | 226 | 46 |
| 2-Chloronaphthalene | 13.0 | 162 | ND | ND | 998 | 566 | 78 |
| Isophorone | 13-15 | 82 | ND | ND | 995 | 525 | 76 |
| Acenaphthylene | 14.3 | 152 | ND | ND | 937 | 528 | 73 |
| Dimethyl phthalate | 16-17 | 163 | ND | ND | 1670 | 957 | 66 |
| 2,6-Dinitrotoluene | 15.9 | 165 | ND | ND | 842 | 450 | 65 |
| 2,4-Dinitrotoluene | 17.1 | 165 | ND | ND | 856 | 487 | 67 |
| Acenaphthene | 14.9 | 154 | ND | ND | 879 | 500 | 69 |
| Fluorene | 16.4 | 166 | ND | ND | 891 | 522 | 71 |
| Diethyl phthalate | 17.6 | 149 | ND | ND | 1680 | 927 | 65 |
| N-nitrosodiphenylamine | 17.6 | 169 | ND | ND | 1400 | 805 | 110 |
| 1,2-Diphenylhydrazine | 17.1 | 77 | ND | ND | 868 | 450 | 66 |
| Hexachlorobenzene | 17.8 | 282 | ND | ND | 933 | 544 | 74 |
| 4-Bromophenyl phenyl ether | 18.1 | 248 | ND | ND | 976 | 541 | 76 |
| Phenanthrene/anthracene | 19.7 | 178 | <100 | <100 | 1840 | 1110 | 73 |
| Di-n-butyl phthalate | 22.0 | 149 | <100 | <100 | 890 | 520 | 68 |
| Fluoranthrene | 23.4 | 202 | <100 | <100 | 854 | 502 | 67 |
| Pyrene | 24.1 | 202 | <100 | <100 | 883 | 508 | 69 |
| Benzidine | 26.0 | 184 | ND | ND | <100 | <100 | <10 |
| Chrysene/benzo(a)anthracene | 28.0 | 228 | <100 | <100 | 1860 | 1010 | 71 |
| Butylbenzyl phthalate | 27.0 | 149 | ND | ND | 925 | 439 | 68 |
| Bis(2-ethylhexyl) phthalate | 27.7 | 149 | 516 | 2170 | 6810 | 10100 | AI |
| Di-n-octyl phthalate | 29.7 | 149 | <100 | <100 | 1100 | 593 | 81 |
| Benzo(b)fluoranthene/ benzo(k)fluoranthene | 31.2 | 252 | ND | <100 | 2700 | 1170 | 96 |
| Benzo(a)pyrene | 32.3 | 252 | ND | <100 | 1590 | 678 | 112 |
| 3,3'-Dichlorobenzidine | 29.3 | 252 | <100 | <100 | 1020 | 548 | 78 |
| Benzo(ghi)perylene | 38.2 | 276 | ND | ND | 2430 | 863 | 165 |
| Indeno(1,2,3-cd)pyrene | 36.7 | 276 | ND | ND | 2200 | 818 | 154 |
| Dibenzo(ah)anthracene | 36.9 | 278 | ND | ND | 2290 | 779 | 151 |

Note: ND = Not detected.
AI = Analytical interference.

## TABLE 19. GC/MS DATA—PERCENT RECOVERY OF SEMIVOLATILE ACID EXTRACTABLE ORGANICS IN FOOD PROCESSING SLUDGE

| Compound | Retention time, min | Ion used for quantitation | Conc found in unspiked samples, μg/L No.1 | Conc found in unspiked samples, μg/L No.2 | Conc found in spiked samples,* μg/L No.1 | Conc found in spiked samples,* μg/L No.2 | Average recovery, % |
|---|---|---|---|---|---|---|---|
| 2-Chlorophenol | 4.7 | 128 | ND | ND | 875 | 983 | 74 |
| 2-Nitrophenol | 5.1 | 139 | ND | ND | 963 | 874 | 73 |
| Phenol | 6.8 | 94 | <250 | <250 | 801 | 702 | 59 |
| 2,4-Dimethylphenol | 8.3 | 122 | ND | ND | <250 | 272 | <20 |
| 2,4-Dichlorophenol | 8.7 | 162 | ND | ND | 1010 | 1020 | 81 |
| 2,4,6-Trichlorophenol | 10.6 | 196 | ND | ND | 1080 | 1070 | 86 |
| 4-Chloro-3-methylphenol | 12.2 | 142 | ND | ND | 927 | 863 | 72 |
| 2,4-Dinitrophenol | 15-16 | 184 | ND | ND | <250 | <250 | <20 |
| Pentachlorophenol | 16.4 | 266 | <250 | <250 | 1330 | 1120 | 96 |
| 4-Nitrophenol | 20.0 | 139/65 | <250 | ND | 1130 | 1210 | 89 |

Note: ND = Not detected.

* Concentration spiked—1250 μg/L.

## TABLE 20. GC/MS DATA—PERCENT RECOVERY OF SEMIVOLATILE BASE/NEUTRAL EXTRACTABLE ORGANICS IN LIME TREATMENT SLUDGE

| Compound | Retention time, min | Ion used for quantitation | Conc found in unspiked samples, µg/L | | Conc found in spiked samples, µg/L | | Average recovery, % |
|---|---|---|---|---|---|---|---|
| | | | No.1 | No.2 | No.1 | No.2 | |
| 1,3-Dichlorobenzene | 3.8 | 146 | ND | ND | 736 | 729 | 73 |
| 1,4-Dichlorobenzene | 4.2 | 146 | ND | ND | 929 | 938 | 93 |
| 1,2-Dichlorobenzene | 4.9 | 146 | ND | ND | 1030 | 1040 | 103 |
| Bis(2-chloroethyl) ether | 6.8 | 93 | ND | ND | 1020 | 1010 | 98 |
| Hexachloroethane | 4.8 | 117 | ND | ND | 840 | 852 | 85 |
| Bis(2-chloroisopropyl) ether | 6-7 | 45 | ND | ND | 953 | 949 | 95 |
| Nitrobenzene | 9.0 | 77 | ND | ND | 1040 | 1150 | 109 |
| Hexachlorobutadiene | 8.5 | 225 | ND | ND | 981 | 963 | 97 |
| Naphthalene | 9.2 | 128 | ND | ND | 1110 | 1080 | 110 |
| 1,2,4-Trichlorobenzene | 9.6 | 180 | ND | ND | 1050 | 1040 | 104 |
| Bis(2-chloroethoxy) methane | 10-12 | 93 | ND | ND | 1050 | 1030 | 104 |
| N-nitrosodipropylamine | 10.5-12.5 | 130/70 | ND | ND | 1060 | 842 | 95 |
| Hexachlorocyclopentadiene | 11.1 | 237 | ND | ND | 766 | 783 | 79 |
| 2-chloronaphthalene | 13.0 | 162 | ND | ND | 976 | 971 | 97 |
| Isophorone | 13-15 | 82 | ND | ND | 912 | 873 | 89 |
| Acenaphthylene | 14.3 | 152 | ND | ND | 933 | 947 | 94 |
| Dimethyl phthalate | 16-17 | 163 | ND | ND | 1300 | 1500 | 70 |
| 2,6-Dinitrotoluene | 15.9 | 165 | ND | ND | 915 | 899 | 91 |
| 2,4-Dinitrotoluene | 17.1 | 165 | <100 | <100 | 917 | 875 | 88 |
| Acenaphthene | 14.9 | 154 | ND | ND | 874 | 872 | 87 |
| Fluorene | 16.4 | 166 | ND | ND | 927 | 931 | 93 |
| Diethyl phthalate | 17.6 | 149 | ND | ND | 1540 | 1580 | 80 |
| N-nitrosodiphenylamine | 17.6 | 169 | ND | ND | 1460 | 1480 | 147 |
| 1,2-Diphenylhydrazine | 17.1 | 77 | ND | ND | 851 | 863 | 86 |
| Hexachlorobenzene | 17.8 | 282 | ND | ND | 911 | 953 | 93 |
| 4-Bromophenyl phenyl ether | 18.1 | 248 | ND | ND | 1060 | 1070 | 106 |
| Phenanthrene/anthracene | 19.7 | 178 | <100 | <100 | 2090 | 2020 | 103 |
| Di-n-butyl phthalate | 22.0 | 149 | <100 | <100 | 966 | 920 | 92 |
| Fluoranthrene | 23.4 | 202 | <100 | <100 | 923 | 830 | 50 |
| Pyrene | 24.1 | 202 | <100 | <100 | 885 | 798 | 84 |
| Benzidine | 26.0 | 184 | ND | ND | <100 | <100 | <10 |
| Chrysene/benzo(a)anthracene | 28.0 | 228 | <100 | ND | 1510 | 1310 | 70 |
| Butylbenzyl phthalate | 27.0 | 149 | ND | ND | 796 | 565 | 68 |
| Bis(2-ethylhexyl) phthalate | 27.7 | 149 | 3560 | 4650 | 1530 | 1760 | <10 |
| Di-n-octyl phthalate | 29.7 | 149 | <100 | <100 | 752 | 495 | 21 |
| Benzo(b)fluoranthene/ benzo(k)fluoranthene | 31.2 | 252 | <100 | ND | 1500 | 1340 | 70 |
| Benzo(a)pyrene | 32.3 | 252 | <100 | ND | 798 | 675 | 72 |
| 3,3'-Dichlorobenzidine | 29.3 | 252 | ND | ND | 914 | 750 | 83 |
| Benzo(ghi)perylene | 38.2 | 276 | <100 | ND | 846 | 747 | 78 |
| Indeno(1,2,3-cd)pyrene | 36.7 | 276 | <100 | ND | 763 | 697 | 71 |
| Dibenzo(ah)anthracene | 36.9 | 278 | <100 | ND | 779 | 664 | 71 |

Note: ND = Not detected.

## TABLE 21. GC/MS DATA—PERCENT RECOVERY OF SEMIVOLATILE ACID EXTRACTABLE ORGANICS IN LIME TREATMENT SLUDGE

| Compound | Retention time, min | Ion used for quantitation | Conc found in unspiked samples, µg/L No.1 | Conc found in unspiked samples, µg/L No.2 | Conc found in spiked samples,* µg/L No.1 | Conc found in spiked samples,* µg/L No.2 | Average recovery, % |
|---|---|---|---|---|---|---|---|
| 2-Chlorophenol | 4.7 | 128 | ND | ND | 871 | 870 | 70 |
| 2-Nitrophenol | 5.1 | 139 | ND | ND | 1080 | 1120 | 88 |
| Phenol | 6.8 | 94 | <250 | <250 | 722 | 717 | 57 |
| 2,4-Dimethylphenol | 8.3 | 122 | ND | ND | <250 | <250 | <20 |
| 2,4-Dichlorophenol | 8.7 | 162 | ND | ND | 886 | 944 | 73 |
| 2,4,6-Trichlorophenol | 10.6 | 196 | ND | ND | 979 | 1110 | 84 |
| 4-Chloro-3-methylphenol | 12.2 | 142 | ND | ND | 808 | 898 | 68 |
| 2,4-Dinitrophenol | 15-16 | 184 | ND | ND | 2270 | 2000 | 171 |
| Pentachlorophenol | 16.4 | 266 | ND | ND | 1440 | 1420 | 114 |
| 4-Nitrophenol | 20.0 | 139/65 | ND | ND | 1650 | 1440 | 124 |

Note: ND = Not detected.

* Concentration spiked—1250 µg/L.

analyze Coke Oven Sludge in conjunction with another program. A summary of the digestion procedures, as well as modifications deemed necessary at that time, are given in Tables 8 through 12.

The results of metal analysis of Coke Oven Sludge are presented in Table 22. These results indicated that all procedures indeed have some limitations. The MRI method required four separate digestions, was extremely time consuming, and appeared to yield low recoveries for several of the metals. Both the BCL and the EMSL-Ci methods required 8 to 10 h for sample preparation with separate digestions for flame and graphite furnace analyses. The EMSL-Ci procedure gave better (higher) recovery values. The UW digestion, however, required only 3 h for completion and gave essentially the same elemental recoveries, except for selenium, as did the EMSL-Ci procedure. The reason for the low selenium recovery, approximately 65% of the concentration obtained by the other procedures, was not determined.

Despite the apparent selenium anomaly, the UW digestion was simple, rapid, and involved the preparation of only one sample for all metal analyses, including mercury. On this basis it appeared to be the method of choice for further evaluation. Some concern was expressed regarding the applicability of the method with samples of high solids content; therefore, at the request of the Task Officer, an additional comparison of the UW and EMSL-Ci procedures was conducted with a Paint Pigment Sludge.

## Comparison of the UW and EMSL-Ci Digestion Procedures with Paint Pigment Sludge

The "as received" Paint Pigment Sludge consisted of a liquid phase and a densely-packed solid phase that was extremely difficult to disperse. A 1-L sample, as representative as could be obtained, was acidified with 300 mL of concentrated $HNO_3$ (Ultrex) to a pH $<2$ and diluted to a volume of 2 L with deionized, distilled water to facilitate ease of handling. A well-shaken sample of the diluted sludge was transferred to a beaker and agitated with a magnetic stirrer while approximately 20 mL aliquots were transferred to pretared ampuls or Erlenmeyer flasks for the respective digestions. Five spiked and unspiked replicates were prepared for each method. Spike levels were selected to give concentrations of approximately 2X the elemental concentration of the unspiked samples. No problems were experienced with any explosions of the sealed ampuls during digestion at 125 °C.

The results of the analyses are given in Table 23 and the results of the spike recoveries in Table 24. As shown in Table 24, the arsenic, antimony and selenium spikes were not recovered from either the UW or EMSL-Ci digestates of the Paint Pigment study. Since essentially complete recovery could be obtained when the spikes were added to the digested, filtered samples, it seems reasonable to assume that significant concentrations of arsenic, antimony and selenium were lost by adsorption of the residue or by the formation of insoluble or slightly soluble compounds.

## TABLE 22. RESULTS OF METAL DETERMINATION IN COKE OVEN SLUDGE[a]

| Element | Midwest Research Institute digestion |  |  | Environmental Monitoring and Support Laboratory digestion |  |  | Battelle-Columbus digestion |  |  | University of Washington digestion |  |  | Standard Environmental Protection Agency digestion |  |  |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
|  | 1 | 2 | 3 | 1 | 2 | 3 | 1 | 2 | 3 | 1 | 2 | 3 | 1 | 2 | 3 |
| Antimony | b | b | b | 12 | 16 | 12 | 13 | 24 | 16 | 10 | 12 | 11 | b | b | - |
| Arsenic | 9000[c] | 11000[c] | 11000[c] | 12000 | 11000 | 11000 | 11000 | 9000 | 11000 | 10000 | 10000 | 10000 | 9400 | 13000 | - |
| Beryllium | <50 | <50 | - | <100 | <100 | <100 | <50 | <50 | <50 | <50 | <50 | <50 | <50 | <50 | - |
| Cadmium | <10 | <10 | - | <20 | <20 | <20 | <10 | <10 | <10 | <10 | <10 | <10 | <10 | <10 | - |
| Chromium | 140 | 140 | - | 450 | 500 | 480 | 460 | 460 | 460 | 620 | 620 | 620 | 470 | 470 | - |
| Copper | 590 | 590 | - | 1100 | 1000 | 1000 | 930 | 940 | 940 | 730 | 910 | 760 | 750 | 720 | - |
| Lead | 940 | 840 | 760 | 1000 | 860 | 880 | 650 | 550 | 800 | 700 | 640 | 760 | 690 | 650 | - |
| Mercury[d] | 200 | 300 | 310 | 340 | 340 | 250 | 260 | 350 | 350 | 390 | 400 | 390 | 450 | 550 | 450 |
| Nickel | 230 | 230 | - | 300 | <300 | <300 | 350 | 350 | 350 | 670 | 670 | 580 | 310 | 230 | - |
| Selenium | 12000[c] | 12000[c] | 13000[c] | 12000 | 12000 | 12000 | 12000 | 11000 | 13000 | 7600[c] | 7600[c] | 7800[c] | 2400 | 6200 | - |
| Silver | 44 | 44 | - | 150 | 100 | 100 | 90 | 80 | 110 | 110 | 110 | 110 | 120 | 120 | - |
| Thallium | <10 | <10 | - | <20 | <20 | <20 | <10 | <10 | <10 | <10 | <10 | <10 | <10 | <10 | - |
| Zinc | 3600 | 3600 | - | 5600 | 5500 | 5500 | 5000 | 4800 | 4700 | 5000 | 5300 | 5100 | 5000 | 5000 | - |

a. All results expressed in micrograms per liter of dilute sludge (1:1 by volume).

b. Severe interference problem with furnace. Foaming could not be controlled in hydride procedure.

c. Hydride procedure.

d. Cold vapor procedure.

## TABLE 23. RESULTS OF METAL DETERMINATION OF PAINT PIGMENT SLUDGE[a]

A. University of Washington digestion

| Element | No. 1 | No. 2 | No. 3 | No. 4 | No. 5 | Mean | Relative standard deviation, % |
|---|---|---|---|---|---|---|---|
| Antimony | <0.10 | <0.10 | <0.10 | <0.10 | <0.10 | <0.10 | - |
| Arsenic | 0.93 | 0.85 | 1.00 | 0.92 | 0.88 | 0.92 | 9.0 |
| Beryllium | 0.04 | 0.02 | 0.02 | 0.02 | 0.02 | 0.02 | 37.3 |
| Cadmium | 0.01 | 0.02 | 0.01 | 0.02 | 0.02 | 0.02 | 34.2 |
| Chromium | 7.7 | 7.3 | 7.2 | 7.7 | 7.6 | 7.5 | 3.1 |
| Copper | 102 | 105 | 103 | 103 | 102 | 103 | 1.2 |
| Lead | 26 | 23 | 22 | 27 | 24 | 24 | 7.5 |
| Mercury[b] | 61 | 57 | 55 | 56 | 57 | 57 | 4.0 |
| Nickel | 1.0 | 1.1 | 1.1 | 1.0 | 1.2 | 1.1 | 8.9 |
| Selenium | <0.05 | <0.05 | <0.05 | <0.05 | <0.05 | <0.05 | - |
| Silver | 0.31 | 0.32 | 0.31 | 0.31 | 0.33 | 0.32 | 2.7 |
| Thallium | <0.02 | <0.02 | <0.02 | <0.02 | <0.02 | <0.02 | - |
| Zinc | 250 | 260 | 260 | 260 | 260 | 260 | 1.9 |

B. Environmental Monitoring and Support Laboratory digestion

| Element | No. 1 | No. 2 | No. 3 | No. 4 | No. 5 | Mean | Relative standard deviation, % |
|---|---|---|---|---|---|---|---|
| Antimony | <0.10 | <0.10 | <0.10 | <0.10 | <0.10 | <0.10 | - |
| Arsenic | 1.3 | 0.88 | 1.0 | 1.2 | 0.96 | 1.1 | 16.4 |
| Beryllium | 0.02 | 0.01 | 0.01 | 0.01 | 0.01 | 0.01 | 37.3 |
| Cadmium | 0.01 | 0.01 | 0.01 | 0.01 | 0.01 | 0.01 | 0.0 |
| Chromium | 10 | 10 | 10 | 10 | 10 | 10 | 0.0 |
| Copper | 103 | 100 | 99 | 102 | 101 | 101 | 1.6 |
| Lead | 36 | 33 | 32 | 34 | 34 | 34 | 4.3 |
| Mercury[b] | 59 | 60 | 62 | 63 | 50 | 59 | 8.8 |
| Nickel | 1.4 | 1.2 | 1.5 | 1.4 | 1.6 | 1.4 | 8.6 |
| Selenium | <0.05 | <0.05 | <0.05 | <0.05 | <0.05 | <0.05 | - |
| Silver | 0.28 | 0.30 | 0.31 | 0.31 | 0.27 | 0.29 | 6.5 |
| Thallium | <0.02 | <0.02 | <0.02 | <0.02 | <0.02 | <0.02 | - |
| Zinc | 290 | 280 | 290 | 300 | 290 | 290 | 1.7 |

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a. All results expressed in micrograms per gram of sludge diluted 1:1 by volume.

b. Mercury was also determined after a standard EPA digestion of this sample. The following results were obtained: 76, 70, 72, 71, and 73 μg/g with an average of 72 μg/g and a % RSD of 32.

## TABLE 24. SPIKE RECOVERY IN PAINT PIGMENT SLUDGE[a]

A. University of Washington digestion

| | Percent recovery | | | | | | |
|---|---|---|---|---|---|---|---|
| Element | No. 1 | No. 2 | No. 3 | No. 4 | No. 5 | Mean | Relative standard deviation, % |
| Antimony | –[b] | –[b] | –[b] | –[b] | –[b] | – | – |
| Arsenic | –[c] | –[c] | –[c] | –[c] | –[c] | – | – |
| Beryllium | 99 | 90 | 98 | 93 | 97 | 95 | 4.0 |
| Cadmium | 101 | 101 | 104 | 105 | 107 | 104 | 2.5 |
| Chromium | 113 | 114 | 112 | 99 | 96 | 107 | 8.0 |
| Copper | 89 | 101 | 99 | 98 | 94 | 96 | 5.0 |
| Lead | 80 | 85 | 96 | 126 | 98 | 97 | 18 |
| Mercury[d] | – | – | – | – | – | – | – |
| Nickel | 96 | 93 | 88 | 93 | 84 | 91 | 5.2 |
| Selenium | –[e] | –[e] | –[e] | –[e] | –[e] | –[e] | –[e] |
| Silver | 29 | 26 | 20 | 21 | 20 | 23 | 17.6 |
| Thallium | 83 | 90 | 74 | 70 | 72 | 78 | 11.0 |
| Zinc | 104 | 133 | 120 | 113 | 103 | 115 | 11.0 |

B. Environmental Monitoring and Support Laboratory digestion

| | Percent recovery | | | | | | |
|---|---|---|---|---|---|---|---|
| Element | No. 1 | No. 2 | No. 3 | No. 4 | No. 5 | Mean | Relative standard deviation, % |
| Antimony | –[b] | –[b] | –[b] | –[b] | –[b] | – | – |
| Arsenic | –[c] | –[c] | –[c] | –[c] | –[c] | – | – |
| Beryllium | 91 | 106 | 96 | 92 | 94 | 96 | 6.3 |
| Cadmium | 100 | 102 | 107 | 112 | 104 | 105 | 4.5 |
| Chromium | 118 | 113 | 101 | 113 | 109 | 111 | 5.7 |
| Copper | 104 | 104 | 99 | 95 | 92 | 99 | 5.4 |
| Lead | 115 | 124 | 112 | 99 | 108 | 112 | 8.2 |
| Mercury[d] | 99 | 108 | 98 | 97 | 84 | 97 | 8.8 |
| Nickel | 95 | 86 | 86 | 96 | 98 | 92 | 6.2 |
| Selenium | –[e] | –[e] | –[e] | –[e] | –[e] | –[e] | –[e] |
| Silver | 71 | 92 | 73 | 73 | 55 | 73 | 17.0 |
| Thallium | 76 | 80 | 106 | 83 | 86 | 86 | 14.0 |
| Zinc | 112 | 118 | 107 | 109 | 104 | 110 | 4.9 |

---

a. Paint Pigment Sludge diluted 1:1 by volume.

b. No recovery of 1.0 µg/g antimony spike.

c. No recovery of 1.0 µg/g arsenic spike.

d. Mercury spikes were inadvertently omitted from the UW and EMSL-Ci samples. These recoveries were obtained from sludge samples spiked only with mercury and then digested by the standard EPA procedures.

e. No recovery of 0.5 µg/g selenium spike.

Although the silver concentration was found to be the same in both the UW and EMSL-Ci unspiked samples, the recovery of silver from the UW spiked series was poor. No satisfactory explanation for this discrepancy was apparent.

In general, the elemental concentrations and spike recovery data obtained from the analyses of the UW digested Paint Pigment samples were essentially the same as those values obtained from the EMSL-Ci digested samples. The relative standard deviations (RSD) were satisfactory for all elements determined to be present at concentrations of 1 μg/g or above.

These results, along with those obtained during the earlier comparison of procedures using the Coke Oven Sludge, indicate the UW procedure to be analytically equivalent or better than the other procedures evaluated. Since the UW procedure is faster (approximately 3 h compared to greater than 8 h for the other procedures) and requires the preparation of only one sample for all metal determinations (three to five samples for other procedures), the UW method was selected as the preferred method for the determination of total metals in residual waste.

# SECTION 5

# EVALUATION OF METHODS

## SUMMARY OF PROPOSED METHODOLOGY

The text of the proposed methods that were subjected to in-depth evaluation for the determination of total content of toxic species in residual wastes is given in Appendix V. The procedures include sample preparation and analysis of purgeable organics by purge-and-trap with GC/MS analysis, base/neutral and acid extractables of semivolatile organics with GC/MS analysis, and acid digestion of metals with AAS analysis. These three methods can be summarized as follows.

> Purgeable organics—An appropriate weight of sludge (determined by a preliminary screening of extractable organic content of the sample) is diluted to 10 mL with organic-free water. The diluted sample is then purged at room temperature (ca. 25 °C) with an inert gas for 12 min. Compounds boiling below 200 °C and of limited solubility in water are transferred from the aqueous phase to the gas phase. The gas phase is passed through a sorbent trap where the organic compounds are concentrated. The contents of the trap are then desorbed into the GC/MS by heating and backflushing the trap. The total analysis time is less than 1 h.
>
> Base/neutral and acid extractable organics—A 40-g sample is extracted with methylene chloride using liquid-liquid extraction techniques aided by a high-speed homogenizer. Samples are extracted at pH $\geq 11$ and again at pH $\leq 2$ to isolate base/neutral and acidic compounds, respectively. The extract is dried with sodium sulfate and concentrated to a volume of 5 mL or less using a Kuderna-Danish (K-D) evaporator. The compounds in the extract are separated, identified and quantified by GC/MS. If interferences are encountered, as is frequently the case, the method provides a general-purpose gel permeation chromatography (GPC) cleanup procedure to eliminate many interferences.
>
> Acid digestion of metals—The sample is transferred to a clean glass ampul, acidified with 2 mL of concentrated $HNO_3$, and diluted to about 20 mL with deionized, distilled water. The ampul is sealed and placed in an oven at 125 °C for 1 h. The digested sample is suction filtered and brought to a final volume of 50 mL with deionized, distilled water. The selected metals are then determined by atomic adsorption spectroscopy using direct aspiration, furnace, gaseous hydride, or cold vapor (mercury) procedures. The use of an ICP spectrometer may also be appropriate.

## DESCRIPTION OF RESIDUAL WASTES

After the preliminary methods evaluations study was completed, several wastes were selected for use in the second phase of the study. Agreement as to which of the available samples would be used was reached in a meeting of the two analytical contractors and EPA-EGD, EPA-OSW, and EMSL-LV personnel. Although the residuals selected for study were limited to nine by time and cost considerations, they were thought to be representative of the wide range of complex matrices that are likely to be encountered in the analysis of wastes. These samples were chosen to include those with high and low liquid organic content, high and low organic and inorganic solids content, and basic and acidic pH values. Several characteristics of the selected waste samples are given in Table 25.

## MATRIX OF ANALYSES

Twelve sets of analyses were completed including the repeat analyses of the Ink Pigment Waste and Organic Still Bottoms. The first set of analyses on these samples was done on the waste as received whereas the second set of analyses (labeled: Interlaboratory) was performed on a sample that was remixed, reproportioned, and resubmitted to the laboratories participating in the interlaboratory validation study. The spiked Method Blank consisted of reagent water spiked with a mixture of the semivolatile organic compounds determined during the initial phase of the program. A summary of the number of replicate analyses and their spike levels for each of the residual wastes in the study is given in Table 26. The analyses were done in five replicates for each residual waste except for the Coke Oven Biological Sludge, Electroplating Sludge, and Electric Furnace Baghouse Dust, which were analyzed in triplicate. A total of 290 individual samples were prepared. This total included 89 for purgeable organics, 109 for for semivolatile organics and 92 for metals. These determinations gave rise to 8908 data points with the following breakdown according to compound type:

| | |
|---|---|
| • Purgeables | 2225 |
| • Base/Neutrals | 4578 |
| • Acids | 1199 |
| • Metals | 906 |

The preliminary evaluation study resulted in over 1000 additional data points for a total in excess of 10,000.

## COMPOUNDS CHOSEN FOR DETERMINATION

The three major classes of compounds that were determined in residual waste samples included 23 purgeable organics, 56 semivolatile organics and 13 metals. The semivolatile organics can be further divided into several subcategories, which included polynuclear aromatic hydrocarbons, chlorinated

## TABLE 25. DESCRIPTION OF RESIDUAL WASTES

| Residual waste | pH | Solid content, % | Category[a] | Physical description of sample |
|---|---|---|---|---|
| Raw POTW | 6.5 | 0.8 | AS | Thin, dark green, aqueous suspension |
| Electroplating Sludge | 9.0 | 25.2 | IS | Dark, wet cake |
| Paint Pigment Sludge (Latex Paint Sludge) | 7.8 | 14.9 | AS | Thick, milky, aqueous suspension |
| Lime Slurry Treatment Sludge | 7.2 | 2.0 | AS | Thin, tan, aqueous suspension |
| Food Processing Sludge | 5.6 | 0.5 | AS | Thin, tan, aqueous suspension |
| Coke Plant Sludge | 9.0 | 3.1 | AS | Dark, aqueous suspension |
| Organic Still Bottoms | 3.6 | 35.9 | OS | Thick, black, chunky, aqueous slurry |
| Baghouse Dust | 6.3 | 99.3 | IS | Fine, dark brown powder |
| Ink Pigment Waste | 11.3 | 14.8 | AS | Pourable, black gel |

[a] AS - aqueous sludge (>80% $H_2O$).
IS - inorganic sludge (>20% inorganic content).
OS - organic sludge (>20% organic content).

## TABLE 26. SUMMARY OF REPLICATES AND SPIKE LEVELS OF PURGEABLE AND SEMIVOLATILE ORGANICS AND METALS FOR THE VALIDATION STUDY[a]

| Residual Waste | Purgeable organics<br>Spike levels[b]<br>0 | <br><br>I | <br><br>II | Semivolatile organics<br>Spike levels[b]<br>0 | <br><br>I | <br><br>II | <br><br>III | Metals<br>Spike levels[b]<br>0 | <br><br>I | <br><br>II |
|---|---|---|---|---|---|---|---|---|---|---|
| POTW Residual Waste | 5 | 5 | 5 | 5 | 5 | 5 | 5 | - | - | - |
| Ink Pigment Waste | - | - | - | 5 | 5 | 5 | - | 5 | 5 | - |
| Organic Still Bottoms | - | - | - | 5 | - | - | - | 5 | 5 | - |
| Ink Pigment Waste (Interlaboratory) | 5 | 5 | 5 | 5 | 3 | 5 | - | 5 | 5 | - |
| Organic Still Bottoms (Interlaboratory | 5 | 5 | 5 | 5 | 3 | 5 | - | 5 | 5 | - |
| Paint Pigment Sludge | 5 | 5 | 5 | 3 | 3 | 3 | - | 5 | 5 | - |
| Coke Oven Biological Sludge | 3 | 3 | 3 | 3 | 3 | 3 | - | - | - | - |
| Ink Pigment Leachate | 5 | 5 | - | 5 | 5 | - | - | 5 | 5 | 5 |
| Organic Still Bottoms Leachate | 5 | 5 | - | 5 | 5 | - | - | 5 | 5 | 5 |
| Electroplating Sludge | - | - | - | - | - | - | - | - | 3 | 3 |
| Electric Furnace Baghouse Dust | - | - | - | - | - | - | - | - | 3 | 3 |
| Spiked Method Blank | - | - | - | - | 5 | - | - | - | - | - |

a. Numbers in the table are the number of replicates of each analysis of a sample-spike combination.

b. Spike level varied for classes of compounds; however, in general, spike levels were between 2X and 20X of the unspiked sample. Zero spike level is the unspiked sample.

hydrocarbons, phthalate esters, chloroalkyl ethers, nitrosamines, benzidines, phenols, and several miscellaneous compounds. The organic compounds were intended to represent a range of boiling points and functionalities, and were chosen from the priority pollutants primarily because of the ready availability of reference standards. The selection of the compounds was also based on available historical information concerning potential composition of industrial wastewaters that were thought to be associated with residual wastes.

## ANALYTICAL INSTRUMENTATION

The analytical finish in the proposed methods depends primarily on a GC/MS for separation, identification, and quantitation of the organic compounds. The requirements for the instrumentation essentially are identical to those set forth in the familiar methods for determination of priority pollutants in wastewater. The methods for the metals were evaluated using an atomic absorption spectrometer, although an ICP spectrometer may be a suitable, and in some instances, a more desirable analytical finish.

## EVALUATION DATA

The analytical and statistical data from the evaluation study for the proposed methodology for total content are given in Appendix IV. These data include the results of each replicate determination of the concentration of each compound; the amount of spike added and the mean percent recovery for each spiked sample, and the calculated mean, standard deviation, and relative standard deviation for each set of replicate analyses. These tabulations are grouped by compound type for each of the nine residual waste samples and for method blanks (spiked distilled, deionized water). Also, typical GC/MS chromatograms, labeled with identified compounds, are presented after each of the tabulations of compound types for each waste sample.

Several observations, interpretations, and conclusions based on these data are summarized and presented in the following section of this report. However, due to the sheer bulk of the data obtained in this study, it is possible that several significant trends or observations might have gone unreported in the initial data assessment that is presented in this document. Future studies of these data should be readily facilitated by the fact that all of the results that were obtained are presented in this report and are also available on computer cards.

# SECTION 6

# STATISTICAL INTERPRETATION OF DATA

## INTRODUCTION

Because a relatively large amount of data (in excess of 10,000 data points) was generated from the laboratory experiments conducted during this study, several statistical interpretations were made to test the validity and significance of these data. It was recognized that these data were not randomly sampled from well-defined populations and that statistical interpretation of these data in a formal inferential manner may not be valid. Therefore, formal inferential statistical analysis of the data was not done; however, the usefulness of the usual descriptive statistical devices such as mean, standard deviation, relative standard deviation, and mean percent recovery was recognized, and these items were included in the empirical treatment of these data.

## SUMMARY OF STATISTICAL TERMS AND FORMULAS

The following statistical terms and formulas were employed in the analyses of the data points:

- Mean—Arithmetic mean or average, defined as the sum of the values of all the observations divided by the number of observations (n).

- Standard deviation—Defined as the positive square root of the variance, which is defined as the sum of squares of the deviations of the observations from the mean ($\bar{x}$) divided by one less than the total number of observations (n-1).

$$\text{std dev} = \sqrt{\frac{\sum_{i=1}^{n} (x_i - \bar{x})^2}{n - 1}}$$

- RSD—Relative standard deviation, defined as the standard deviation divided by the mean and multiplied by 100.

$$\text{RSD, \%} = \frac{\text{std dev}}{\text{mean}} \times 100$$

- Mean percent recovery—Defined as 100 times the ratio of the difference between the mean concentration of the spiked samples ($\bar{x}_{LI}$) and mean concentration of the unspiked sampies ($\bar{x}_{LO}$) to the amount added.

$$\bar{p} = \left(\frac{\bar{x}_{LI} - \bar{x}_{LO}}{\text{amount added}}\right)(100)$$

## REJECTION OF AN OBSERVATION

Suspected outliers among the data points were tested for rejection in this study by application of the Dixon Criteria for Testing[1] (Q-Test, 90% confidence). A total of only 1.1% of all of the data (1.2% of the data for organics and 0.1% of the data for metals) was found to be suspect and these outliers were not included in the other statistical calculations.

## INTERQUARTILE RANGES

A very useful means for summarizing the significance of the statistical results of the method evaluation study involved the use of the so-called interquartile range.[2] This range is defined by those results, ranked according to magnitude, that occur in the middle one-half of the ranking order (i.e., data ranked within 25 to 75% of the total number of data points). Thus, 50% of the results will occur in this median range, and it was the use of this range that formed the basis of the conclusions presented in Section 2 of this report. These interquartile ranges of the RSD and mean percent recoveries are given in Tables 27 and 28 for each residual waste as a function of the various compound groups. It was concluded from these results that the proposed methods for total content could be applied to the analysis of a typical residual waste with a reasonable expectation that an accuracy of approximately -50 to 150% of the true values and a precision of approximately 10 to 25% RSD for organics and 3 to 7% for metals can be obtained.

Otherwise, the summaries of the interquartile ranges indicated that there was not a great deal of difference among the methods for the various classes of organics with regard to accuracy and precision. On the other hand, RSD for determination of metals (by AAS) was substantially smaller than that for determination of organics. Accuracy of each of the methods (based on spike recoveries) was similar, in general, with no clear trend emerging.

Also included in the summary tables is a treatment of the data from the interlaboratory validation studies of the potential mobility (leachate) methods and the total content methods. The detailed presentation of the interlaboratory study data is made in Appendices II and III. These results are not discussed in detail here, but are provided for future comparison with the results obtained by BCL in the analysis of nominally-identical samples by the same methodologies.

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1. Dixon, W. J., "Processing Data for Outliers," Biometrics 9(1), 74-89, 1953.

2. David, H. A., Order Statistics, John Wiley and Son, Inc., New York, NY, 1970.

TABLE 27. SUMMARY OF INTERQUARTILES OF RELATIVE STANDARD DEVIATIONS OF CONCENTRATIONS OF COMPOUNDS AVERAGED OVER ALL SPIKE LEVELS (INCLUDING UNSPIKED SAMPLES)[a]

| | | Total content | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | Residual waste | | | | | | | | | Potential mobility | |
| Class | Range | POTW | IPW | OSB | PPS | COBS | EPS | EFBHD | IPW-IL | OSB-IL | IPL | OSBL |
| Base/Neutrals | high | 25 | 20 | 33 | 19 | 16 | - | - | 29 | 31 | 25 | 22 |
| | low | 19 | 12 | 25 | 7 | 8 | - | - | 19 | 19 | 10 | 14 |
| Acids | high | 34 | 44 | 17 | 31 | 20 | - | - | 21 | 29 | 18 | 19 |
| | low | 23 | 27 | 0 | 6 | 13 | - | - | 24 | 10 | 10 | 15 |
| Purgeables | high | 24 | - | - | 14 | 15 | - | - | 26 | 20 | 22 | 22 |
| | low | 11 | - | - | 8 | 10 | - | - | 15 | 16 | 10 | 12 |
| Metals | high | - | 6 | 10 | 5 | - | 5 | 6 | 5 | 10 | 3 | 3 |
| | low | - | 4 | 7 | 3 | - | 2 | 3 | 3 | 3 | 1 | 2 |

a. Relative standard deviations in this table are expressed as percent (%).

## TABLE 28. SUMMARY OF INTERQUARTILES OF AVERAGE PERCENT RECOVERIES OF COMPOUNDS IN SPIKED RESIDUAL WASTE SAMPLES[a]

| | | Total content | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | Spiked method blank | Residual waste | | | | | | | | | Potential mobility | |
| Class | Range | | POTW | IPW | OSB | PPS | COBS | EPS | EFBHD | IPW-IL | OSB-IL | IPL | OSBL |
| Base/Neutrals | high | 109 | 155 | 132 | - | 53 | 84 | - | - | 88 | 115 | 95 | 147 |
| | low | 70 | 72 | 78 | - | 21 | 59 | - | - | 57 | 63 | 67 | 72 |
| Acids | high | 76 | 70 | 102 | - | 48 | 119 | - | - | 122 | 69 | 102 | 41 |
| | low | 55 | 53 | 70 | - | 11 | 109 | - | - | 109 | 21 | 78 | 25 |
| Purgeables | high | - | 70 | - | - | 147 | 95 | - | - | 52 | 98 | 81 | 32 |
| | low | - | 51 | - | - | 77 | 75 | - | - | 24 | 87 | 54 | 17 |
| Metals | high | - | - | 102 | 94 | 99 | - | 96 | 94 | 106 | 104 | 106 | 108 |
| | low | - | - | 81 | 74 | 69 | - | 64 | 71 | 98 | 82 | 100 | 99 |

a. Recoveries in this table are expressed as percent (%).

## SUMMARY PRESENTATION OF DATA

A summary of the average concentrations of all compounds determined in the various (unspiked) residual waste samples is presented in Appendix I following this section. Tables I-1 through I-4 give these data separately for the following compound groups respectively: purgeable organics, base/neutral fraction of semivolatiles, acid fraction of semivolatiles, and metals. These data are the mean results of replicate analyses for the various waste samples. The key at the beginning of this group of tables should be referred to for identification of the abbreviations used for the various waste samples.

A brief review of these analytical results indicates the diversity and complexity of these sample matrices.

Tables I-5 through I-8 in Appendix I present the average relative standard deviations (RSD) for each determination in each sample matrix. These data were obtained from the RSD for each compound determination at all spike levels (including unspiked). These results are also presented separately for compound groups and are a measure of the analytical precision of the methods for the various compounds.

The averages of the mean percent recoveries of spikes for each compound in the various sample matrices are given in Tables I-9 through I-12. These data are also presented for each compound group and represent a measure of accuracy of the methods. In some isolated cases, inordinately high values (e.g., >1000%) or values less than 0% for recovery were obtained for some compounds. These results were almost always caused by the spike level being much smaller than the concentration of the compound in the unspiked sample. Appropriate spiking techniques called for spiking at 2, 10, and 20 times the unspiked concentrations in the various samples. However, out of necessity, the spiked and unspiked samples were occasionally analyzed simultaneously, so this procedure was not always followed.

# Appendix I. Data Summary Tables

# Legend for Verification Analyses
# and Quality Assurance
# for Total Content Data*

| Symbols | Identification |
|---|---|
| . | Not determined. |
| < | Lower detection limit of instrumentation. |
| POTW | POTW Residual Waste. |
| IPW | Ink Pigment Waste. |
| PPS | Paint Pigment Sludge. |
| COBS | Coke Oven Biological Sludge. |
| EPS | Electroplating Sludge. |
| EFBHD | Electric Furnace Baghouse Dust. |
| IPW-IL | Ink Pigment Waste (Interlaboratory). |
| OSB-IL | Organic Still Bottoms (Interlaboratory). |
| IPL | Ink Pigment Leachate. |
| OSBL | Organic Still Bottoms Leachate. |

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* Concentrations are reported as microgram of compound per gram of waste (parts per million).

# A. Summary of Average Concentrations of Compounds in Unspiked Residual Waste Samples

## TABLF I-1. AVERAGE CONCENTRATIONS OF PURGEABLE ORGANICS IN UNSPIKED RESIDUAL WASTE SAMPLES

| COMPOUND | PGTW | IPW | OSB | PPS | CDBS | EPS | EPBMD | IPW_IL | OSB_IL | IPL | OSBL |
|---|---|---|---|---|---|---|---|---|---|---|---|
| METHYLENE CHLORIDE | 0.66 | . | . | 0.74 | 0.09 | . | . | 6.0 | 54.8 | 1201.2 | 759 |
| TRICHLOROFLUOROMETHANE | . | . | . | . | . | . | . | . | 0.0 | . | . |
| 1,1-DICHLOROETHYLENE | 0.00 | . | . | 0.00 | 0.00 | . | . | 0.7 | 93.0 | 74.2 | 7348 |
| 1,1-DICHLOROETHANE | 0.00 | . | . | 0.00 | 0.00 | . | . | 0.0 | 2168.0 | [illegible] | 170600 |
| TRANS-1,2-DICHLOROETHYLENE | 0.00 | . | . | 0.00 | 0.00 | . | . | 0.0 | [illegible] | [illegible] | [illegible] |
| CHLOROFORM | 0.00 | . | . | 0.00 | 0.00 | . | . | 0.0 | [illegible] | 0.0 | [illegible] |
| 1,2-DICHLOROETHANE | 0.00 | . | . | 0.00 | 0.00 | . | . | 0.0 | 1496.0 | 0.0 | [illegible] |
| 1,1,1-TRICHLOROETHANE | 0.00 | . | . | 0.00 | 0.00 | . | . | 0.0 | 354.8 | [illegible] | 23740 |
| CARBON TETRACHLORIDE | 0.00 | . | . | 0.00 | 0.00 | . | . | 0.0 | 59.8 | 0.0 | 0 |
| BROMODICHLOROMETHANE | 0.00 | . | . | 0.00 | 0.00 | . | . | 0.0 | 0.0 | 0.0 | 0 |
| 1,2-DICHLOROPROPANE | 0.00 | . | . | 0.00 | 0.00 | . | . | 0.0 | 0.0 | 0.0 | 0 |
| TRANS-1,3-DICHLOROPROPENE | 0.00 | . | . | 0.00 | 0.00 | . | . | 0.0 | 0.0 | 0.0 | 0 |
| TRICHLOROETHYLENE | 0.00 | . | . | [illegible] | 0.00 | . | . | 0.0 | 577.0 | 76.0 | 14960 |
| DIBROMOCHLOROMETHANE | 0.00 | . | . | 0.00 | 0.00 | . | . | 0.0 | 0.0 | 0.0 | 0 |
| CIS-1,3-DICHLOROPROPENE | 0.00 | . | . | 0.00 | 0.00 | . | . | 0.0 | 0.0 | 0.0 | 0 |
| 1,1,2-TRICHLOROETHANE | 0.00 | . | . | 0.00 | 0.00 | . | . | 0.0 | 7104.0 | 0.0 | 26760 |
| BENZENE | 0.00 | . | . | [illegible] | 0.00 | . | . | 0.0 | 0.0 | [illegible] | 0 |
| BROMOFORM | 0.00 | . | . | 0.00 | 0.00 | . | . | 0.0 | 0.0 | 0.0 | 0 |
| 1,1,2,2-TETRACHLOROETHENE | 0.00 | . | . | [illegible] | 0.00 | . | . | [illegible] | [illegible] | [illegible] | [illegible] |
| 1,1,2,2-TETRACHLOROETHANE | 0.00 | . | . | [illegible] | 0.00 | . | . | [illegible] | [illegible] | [illegible] | [illegible] |
| TOLUENE | [illegible] | . | . | 14.00 | 0.00 | . | . | [illegible] | [illegible] | [illegible] | [illegible] |
| CHLOROBENZENE | [illegible] | . | . | 2.07 | 0.00 | . | . | [illegible] | [illegible] | 0.0 | 0 |
| ETHYLBENZENE | 1.00 | . | . | 250.67 | 0.00 | . | . | 57.2 | 0.0 | 1720.0 | 0 |

TABLE I-2. AVERAGE CONCENTRATIONS OF BASE/NEUTRAL EXTRACTABLE SEMIVOLATILE ORGANICS IN UNSPIKED RESIDUAL WASTE SAMPLES

| COMPOUND | PCIW | IPW | D10 | PPS | COBS | EPS | EPBND | IPW_IL | D10_IL | IPL | D10L |
|---|---|---|---|---|---|---|---|---|---|---|---|
| 1,3-DICHLOROBENZENE | 2.00 | 0.0 | 17.0 | 0.00 | 0.00 | . | . | 0.0 | [illegible] | 0.0 | [illegible] |
| 1,4-DICHLOROBENZENE | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | . | . | [illegible] | [illegible] | [illegible] | [illegible] |
| 1,2-DICHLOROBENZENE | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | . | . | [illegible] | [illegible] | [illegible] | [illegible] |
| HEXACHLOROETHANE | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | . | . | [illegible] | [illegible] | [illegible] | [illegible] |
| BIS(2-CHLOROETHYL) ETHER | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | . | . | [illegible] | [illegible] | [illegible] | [illegible] |
| BIS(2-CHLOROISOPROPYL) ETHER | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | . | . | [illegible] | [illegible] | [illegible] | [illegible] |
| HEXACHLOROBUTADIENE | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | . | . | [illegible] | [illegible] | [illegible] | [illegible] |
| NITROBENZENE | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | . | . | [illegible] | [illegible] | [illegible] | [illegible] |
| NAPHTHALENE | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | . | . | [illegible] | [illegible] | [illegible] | [illegible] |
| 1,2,4-TRICHLOROBENZENE | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | . | . | [illegible] | [illegible] | [illegible] | [illegible] |
| BIS(2-CHLOROETHOXY)METHANE | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | . | . | [illegible] | [illegible] | [illegible] | [illegible] |
| N-NITROSODI-N-PROPYLAMINE | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | . | . | [illegible] | [illegible] | [illegible] | [illegible] |
| HEXACHLOROCYCLOPENTADIENE | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | . | . | [illegible] | [illegible] | [illegible] | [illegible] |
| 2-CHLORONAPHTHALENE | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | . | . | [illegible] | [illegible] | [illegible] | [illegible] |
| ISOPHORONE | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | . | . | [illegible] | [illegible] | [illegible] | [illegible] |
| ACENAPHTHYLENE | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | . | . | [illegible] | [illegible] | [illegible] | [illegible] |
| ACENAPHTHENE | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | . | . | [illegible] | [illegible] | [illegible] | [illegible] |
| DIMETHYL PHTHALATE | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | . | . | [illegible] | [illegible] | [illegible] | [illegible] |
| 2,6-DINITROTOLUENE | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | . | . | [illegible] | [illegible] | [illegible] | [illegible] |
| FLUORENE | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | . | . | [illegible] | [illegible] | [illegible] | [illegible] |
| 2,4-DINITROTOLUENE | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | . | . | [illegible] | [illegible] | [illegible] | [illegible] |
| 1,2-DIPHENYLHYDRAZINE | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | . | . | [illegible] | [illegible] | [illegible] | [illegible] |
| 4-CHLOROPHENYL PHENYL ETHER | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | . | . | [illegible] | [illegible] | [illegible] | [illegible] |
| DIETHYL PHTHALATE | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | . | . | [illegible] | [illegible] | [illegible] | [illegible] |
| N-NITROSODIPHENYLAMINE | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | . | . | [illegible] | [illegible] | [illegible] | [illegible] |
| HEXACHLOROBENZENE | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | . | . | [illegible] | [illegible] | [illegible] | [illegible] |
| 4-BROMOPHENYL PHENYL ETHER | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | . | . | [illegible] | [illegible] | [illegible] | [illegible] |
| PHENANTHRENE/ANTHRACENE | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | . | . | [illegible] | [illegible] | [illegible] | [illegible] |
| DI-N-BUTYL PHTHALATE | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | . | . | [illegible] | [illegible] | [illegible] | [illegible] |
| FLUORANTHENE | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | . | . | [illegible] | [illegible] | [illegible] | [illegible] |
| PYRENE | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | . | . | [illegible] | [illegible] | [illegible] | [illegible] |
| BENZIDINE | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | . | . | [illegible] | [illegible] | [illegible] | [illegible] |
| BUTYLBENZYL PHTHALATE | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | . | . | [illegible] | [illegible] | [illegible] | [illegible] |
| BIS(2-ETHYLHEXYL) PHTHALATE | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | . | . | [illegible] | [illegible] | [illegible] | [illegible] |
| CHRYSENE/BENZ(A)ANTHRACENE | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | . | . | [illegible] | [illegible] | [illegible] | [illegible] |
| 3,3'-DICHLOROBENZIDINE | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | . | . | [illegible] | [illegible] | [illegible] | [illegible] |
| DI-N-OCTYL PHTHALATE | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | . | . | [illegible] | [illegible] | [illegible] | [illegible] |
| BENZO(B)/BENZO(K)FLUORANTHENES | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | . | . | [illegible] | [illegible] | [illegible] | [illegible] |
| BENZO(A)PYRENE | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | . | . | [illegible] | [illegible] | [illegible] | [illegible] |
| INDENO(1,2,3-CD)PYRENE | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | . | . | [illegible] | [illegible] | [illegible] | [illegible] |
| DIBENZ(A,H)ANTHRACENE | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | . | . | [illegible] | [illegible] | [illegible] | [illegible] |
| BENZO(GHI)PERYLENE | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | . | . | [illegible] | [illegible] | [illegible] | [illegible] |

## TABLE I-3. AVERAGE CONCENTRATIONS OF ACID EXTRACTABLE SEMIVOLATILE ORGANICS IN UNSPIKED RESIDUAL WASTE SAMPLES

| COMPOUND | PGT* | IPW | GSB | PPS | CCDS | EPS | EPGMD | IPW_IL | GSB_IL | IPL | GSBL |
|---|---|---|---|---|---|---|---|---|---|---|---|
| 2-CHLOROPHENOL | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | * | * | [illegible] | [illegible] | [illegible] | [illegible] |
| 2-NITROPHENOL | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | * | * | [illegible] | [illegible] | [illegible] | [illegible] |
| PHENOL | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | * | * | [illegible] | [illegible] | [illegible] | [illegible] |
| 2,4-DIMETHYLPHENOL | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | * | * | [illegible] | [illegible] | [illegible] | [illegible] |
| 2,4-DICHLOROPHENOL | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | * | * | [illegible] | [illegible] | [illegible] | [illegible] |
| 2,4,6-TRICHLOROPHENOL | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | * | * | [illegible] | [illegible] | [illegible] | [illegible] |
| 4-CHLORO-3-METHYLPHENOL | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | * | * | [illegible] | [illegible] | [illegible] | [illegible] |
| 2,4-DINITROPHENOL | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | * | * | [illegible] | [illegible] | [illegible] | [illegible] |
| 4,6-DINITRO-O-CRESOL | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | * | * | [illegible] | [illegible] | [illegible] | [illegible] |
| PENTACHLOROPHENOL | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | * | * | [illegible] | [illegible] | [illegible] | [illegible] |
| 4-NITROPHENOL | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | * | * | [illegible] | [illegible] | [illegible] | [illegible] |

## TABLE I-4. AVERAGE CONCENTRATIONS OF METALS IN UNSPIKED RESIDUAL WASTE SAMPLES

| ELEMENT | POTW | IPW | OSB | PPS | COBS | EPS | EFBHD | IPW_IL | OSB_IL | IPL | OSBL |
|---|---|---|---|---|---|---|---|---|---|---|---|
| ANTIMONY | . | 0.3 | <0.1 | <0.10 | . | 6.9 | 8.7 | . | . | . | . |
| ARSENIC | . | 0.9 | 1.0 | 1.66 | . | 3.0 | 34.0 | . | . | . | . |
| BERYLLIUM | . | <0.1 | <0.1 | <0.10 | . | <0.3 | <0.2 | <0.2 | <0.2 | . | . |
| CADMIUM | . | <0.2 | <0.2 | <0.10 | . | 0.6 | 9.2 | <0.2 | <0.2 | 7.2 | 13 |
| CHROMIUM | . | 77.8 | 37.0 | 13.60 | . | 2500.0 | 1600.0 | 55.4 | 39.0 | 340.2 | 21 |
| CCPPER | . | 10.3 | 627.4 | 185.40 | . | 25100.0 | 423.0 | 9.6 | 884.0 | <50.0 | 23640 |
| LEAD | . | 311.2 | 10.5 | 44.00 | . | 1260.0 | 1320.0 | 240.6 | 21.6 | 7540.0 | 268 |
| MERCURY | . | <0.1 | 0.8 | 103.20 | . | <0.0 | 1.3 | . | . | . | . |
| NICKEL | . | 1.0 | 104.0 | 1.96 | . | 4033.3 | 547.7 | 1.7 | 91.2 | 64.0 | 1448 |
| SELENIUM | . | <0.5 | <0.7 | <0.10 | . | <1.0 | <1.0 | . | . | . | . |
| SILVER | . | <0.1 | 0.3 | 0.60 | . | <0.5 | 0.8 | <0.2 | <0.2 | . | . |
| THALLIUM | . | <0.5 | <0.3 | <0.10 | . | <1.0 | <1.0 | <1.0 | <1.0 | . | . |
| ZINC | . | 29.4 | 55.3 | 464.40 | . | 1906.7 | 68100.0 | 26.2 | 222.4 | 1562.0 | 5790 |

## B. Summary of Relative Standard Deviations of Concentrations of Compounds Averaged Over All Spike Levels (Including Unspiked Samples)

TABLE I-5. RELATIVE STANDARD DEVIATIONS OF CONCENTRATIONS OF PURGEABLE ORGANICS AVERAGED OVER ALL SPIKE LEVELS (INCLUDING UNSPIKED SAMPLES)

| COMPOUND | PCTW | IPW | GSB | PPS | COBS | EPS | EFBHD | IPW_IL | OSB_IL | IPL | OSBL |
|---|---|---|---|---|---|---|---|---|---|---|---|
| METHYLENE CHLORIDE | 37.7 | . | . | 27.2 | 23.2 | . | . | 24.2 | 35.5 | 18.0 | 6.2 |
| TRICHLOROFLUORCMETHANE | . | . | . | . | . | . | . | . | 22.0 | . | . |
| 1,1-DICHLOROETHYLENE | 16.5 | . | . | 6.7 | 12.4 | . | . | 25.7 | 20.9 | 11.4 | 16.7 |
| 1,1-DICHLOROETHANE | 12.8 | . | . | 1.3 | 12.8 | . | . | 15.1 | 19.6 | 22.0 | 11.8 |
| TRANS-1,2-DICHLOROETHYLENE | 23.4 | . | . | 6.0 | 10.0 | . | . | 19.3 | 20.5 | 7.5 | 13.5 |
| CHLOROFORM | 10.9 | . | . | 3.8 | 10.2 | . | . | 19.0 | 18.4 | 8.9 | 15.0 |
| 1,2-DICHLOROETHANE | 33.6 | . | . | 10.4 | 9.2 | . | . | 2.8 | 15.7 | 2.4 | 13.8 |
| 1,1,1-TRICHLOROETHANE | 12.0 | . | . | 6.7 | 11.4 | . | . | 15.6 | 19.5 | 18.6 | 17.9 |
| CARBON TETRACHLORIDE | 24.1 | . | . | 9.0 | 11.5 | . | . | 22.6 | 26.0 | 26.5 | 34.1 |
| BROMODICHLOROMETHANE | 19.4 | . | . | 8.6 | 10.1 | . | . | 16.5 | 18.8 | . | . |
| 1,2-DICHLCROPROPANE | 11.1 | . | . | 8.5 | 16.6 | . | . | 9.3 | 18.5 | . | . |
| TRANS-1,3-DICHLOROPROPENE | 25.7 | . | . | 9.2 | 18.7 | . | . | 15.2 | 15.9 | . | . |
| TRICHLORGETHYLENE | 19.5 | . | . | 32.8 | 12.4 | . | . | 24.0 | 18.4 | 10.3 | 9.2 |
| DIBROMOCHLORCMETHANE | 11.0 | . | . | . | 12.3 | . | . | 2.9 | 17.8 | . | . |
| CIS-1,3-DICHLOROPROPENE | 40.3 | . | . | . | 15.1 | . | . | 13.6 | 11.5 | . | . |
| 1,1,2-TRICHLOROETHANE | 10.4 | . | . | . | 7.9 | . | . | 6.1 | 11.2 | 11.0 | 5.9 |
| BENZENE | 15.1 | . | . | 12.0 | 20.5 | . | . | 21.3 | 14.9 | 11.6 | 30.0 |
| BROMOFORM | 9.4 | . | . | 13.8 | 11.4 | . | . | 32.7 | 18.4 | . | . |
| 1,1,2,2-TETRACHLORGETHENE | 22.8 | . | . | 13.2 | 12.2 | . | . | 27.7 | 12.3 | 30.1 | 16.3 |
| 1,1,2,2-TETRACHLORGETHANE | 25.7 | . | . | 15.0 | 12.0 | . | . | 33.2 | 28.2 | 29.9 | 13.5 |
| TOLUENE | 13.2 | . | . | 10.8 | 16.6 | . | . | 25.3 | 17.1 | 5.9 | 27.1 |
| CHLOROBENZENE | 16.2 | . | . | 25.5 | 16.8 | . | . | 29.7 | 13.8 | 21.4 | 22.4 |
| ETHYLBENZENE | 10.1 | . | . | 18.5 | 8.7 | . | . | 34.6 | 17.8 | 22.6 | 29.3 |

TABLE I-6. RELATIVE STANDARD DEVIATIONS OF CONCENTRATIONS OF BASE/NEUTRAL EXTRACTABLE SEMIVOLATILE ORGANICS AVERAGED OVER ALL SPIKE LEVELS (INCLUDING UNSPIKED SAMPLES)

| COMPOUND | POTW | IPW | OSB | PPS | COBS | EPS | EFBHD | IPW_IL | OSB_IL | IPL | OSBL |
|---|---|---|---|---|---|---|---|---|---|---|---|
| 1,3-DICHLOROBENZENE | 21.5 | 43.0 | 27.7 | . | 6.7 | . | . | 19.1 | 19.9 | . | . |
| 1,4-DICHLOROBENZENE | 22.3 | 11.6 | 19.6 | . | 19.2 | . | . | 26.7 | 29.4 | 24.8 | 17.3 |
| 1,2-DICHLOROBENZENE | 18.0 | 12.2 | 36.6 | . | 9.3 | . | . | 26.0 | 23.9 | 21.3 | 5.6 |
| HEXACHLOROETHANE | 23.2 | 11.0 | 9.3 | 26.6 | 8.1 | . | . | 21.0 | 26.9 | 29.0 | . |
| BIS(2-CHLOROETHYL) ETHER | 54.3 | 19.0 | . | 42.4 | 11.2 | . | . | 47.2 | 20.7 | 15.4 | 18.6 |
| BIS(2-CHLOROISOPROPYL) ETHER | 36.4 | 23.5 | . | 19.3 | 24.3 | . | . | 37.4 | 20.6 | 11.9 | 9.2 |
| HEXACHLOROBUTADIENE | 14.4 | 12.2 | 31.1 | 4.9 | 14.4 | . | . | 10.7 | 26.2 | 13.5 | 21.0 |
| NITROBENZENE | 23.5 | 13.7 | . | 14.8 | 25.0 | . | . | 60.0 | 61.5 | 20.6 | 15.4 |
| NAPHTHALENE | 19.1 | 7.1 | . | 11.0 | 15.4 | . | . | 24.9 | 25.1 | 34.4 | . |
| 1,2,4-TRICHLOROBENZENE | 17.9 | 12.6 | 25.2 | 30.9 | 13.1 | . | . | 24.3 | 21.2 | 13.4 | 13.5 |
| BIS(2-CHLOROETHOXY)METHANE | 30.2 | 12.3 | . | 21.5 | 18.7 | . | . | 21.8 | 8.7 | 11.8 | . |
| N-NITROSODI-N-PROPYLAMINE | 30.6 | 14.5 | . | 15.4 | 43.1 | . | . | 33.7 | 24.6 | 12.9 | . |
| HEXACHLOROCYCLOPENTADIENE | . | . | . | . | 10.8 | . | . | 48.6 | 71.9 | 6.7 | 36.0 |
| 2-CHLORONAPHTHALENE | 20.8 | 12.6 | . | 15.7 | 14.6 | . | . | 22.1 | 17.2 | . | . |
| ISOPHORONE | 20.1 | 24.4 | 16.5 | 13.6 | 19.7 | . | . | 17.7 | 34.3 | 14.4 | 14.5 |
| ACENAPHTHYLENE | 18.9 | 11.2 | 26.1 | 14.7 | 14.3 | . | . | 34.2 | 41.9 | 19.2 | 8.2 |
| ACENAPHTHENE | 16.1 | 11.5 | . | 10.4 | 9.6 | . | . | 25.5 | 29.3 | 24.6 | . |
| DIMETHYL PHTHALATE | 22.1 | 11.8 | . | 9.8 | 7.4 | . | . | 30.0 | 18.1 | 32.9 | 12.8 |
| 2,6-DINITROTOLUENE | 27.5 | 11.9 | . | . | 20.3 | . | . | 16.9 | 21.0 | 13.6 | 33.3 |
| FLUORENE | 19.4 | 12.3 | . | 3.3 | 13.4 | . | . | 24.2 | 44.6 | 13.2 | 12.5 |
| 2,4-DINITROTOLUENE | 39.6 | 15.2 | . | 14.1 | 17.1 | . | . | 16.1 | 33.3 | 47.5 | 23.7 |
| 1,2-DIPHENYLHYDRAZINE | 20.0 | 21.7 | . | 19.6 | 10.5 | . | . | 11.2 | 43.1 | 38.9 | 31.6 |
| 4-CHLOROPHENYL PHENYL ETHER | 24.8 | 16.2 | 28.4 | 7.1 | 11.1 | . | . | 14.1 | 17.9 | 24.2 | 14.1 |
| DIETHYL PHTHALATE | 23.0 | 23.3 | . | 18.4 | 8.7 | . | . | 25.6 | 31.1 | 31.1 | 43.2 |
| N-NITROSODIPHENYLAMINE | 31.2 | 32.9 | . | 26.2 | 8.0 | . | . | 15.0 | 50.1 | . | . |
| HEXACHLOROBENZENE | 22.7 | 16.3 | 33.0 | 14.1 | 24.3 | . | . | 35.5 | 18.7 | 4.9 | 16.0 |
| 4-BROMOPHENYL PHENYL ETHER | 28.0 | 34.9 | . | 14.8 | 11.4 | . | . | 30.8 | 17.5 | 6.8 | 15.8 |
| PHENANTHRENE/ANTHRACENE | 13.4 | 11.4 | . | 9.4 | 6.8 | . | . | 25.0 | 40.8 | 4.4 | . |
| DI-N-BUTYL PHTHALATE | 20.6 | 19.6 | . | 20.6 | 9.3 | . | . | 27.2 | 14.8 | 8.9 | 20.2 |
| FLUORANTHENE | 19.7 | 12.4 | . | 25.8 | 8.3 | . | . | 23.1 | 52.0 | 10.2 | 12.5 |
| PYRENE | 19.0 | 11.7 | . | 14.0 | 8.5 | . | . | 24.0 | 28.6 | 11.8 | 16.6 |
| BENZIDINE | 23.4 | 30.2 | . | . | 22.0 | . | . | 24.5 | 50.0 | . | 21.5 |
| BUTYLBENZYL PHTHALATE | 20.6 | 49.4 | . | 19.9 | 11.2 | . | . | 17.0 | 18.6 | . | . |
| BIS(2-ETHYLHEXYL) PHTHALATE | 19.9 | 9.2 | 46.7 | 19.1 | 14.6 | . | . | 12.0 | 26.1 | 124.4 | 18.2 |
| CHRYSENE/BENZO(A)ANTHRACENE | 20.0 | 18.0 | 70.3 | 20.2 | 11.0 | . | . | 21.5 | 37.9 | 6.8 | 18.9 |
| 3,3'-DICHLOROBENZIDINE | 22.1 | 48.5 | . | 23.9 | 16.9 | . | . | 65.9 | 12.9 | 10.4 | 50.2 |
| DI-N-OCTYL PHTHALATE | 29.8 | . | . | 23.7 | . | . | . | 33.7 | 20.1 | 61.1 | 19.4 |
| BENZO(B)/BENZO(K)FLUORANTHENES | 18.6 | 14.8 | . | 15.6 | 12.3 | . | . | 29.1 | 17.4 | 9.7 | 23.7 |
| BENZO(A)PYRENE | 22.4 | 15.1 | . | 7.9 | 14.8 | . | . | 24.9 | 20.0 | . | . |
| INDENO(1,2,3-CD)PYRENE | 19.7 | 21.0 | . | . | 6.6 | . | . | 84.9 | 3.4 | . | . |
| DIBENZO(AH)ANTHRACENE | 25.1 | 19.0 | . | . | . | . | . | . | 49.5 | . | . |
| BENZO(GHI)PERYLENE | 21.0 | 15.4 | . | 16.2 | 16.4 | . | . | 26.3 | 32.2 | . | . |

TABLE I-7. RELATIVE STANDARD DEVIATIONS OF CONCENTRATIONS OF ACID EXTRACTABLE SEMIVOLATILE ORGANICS AVERAGED OVER ALL SPIKE LEVELS (INCLUDING UNSPIKED SAMPLES)

| COMPOUND | POTW | IPW | OSB | PPS | COBS | EPS | EFBHD | IPW_IL | OSB_IL | IPL | OSBL |
|---|---|---|---|---|---|---|---|---|---|---|---|
| 2-CHLOROPHENOL | 22.6 | 28.2 | . | 30.6 | 10.6 | . | . | 28.3 | 35.8 | 8.8 | 17.6 |
| 2-NITROPHENOL | 32.8 | 22.9 | . | . | 10.9 | . | . | 24.5 | 5.4 | . | . |
| PHENOL | 19.5 | 15.7 | 17.2 | 30.9 | 16.0 | . | . | 10.0 | 10.8 | 9.3 | 13.8 |
| 2,4-DIMETHYLPHENOL | 38.0 | 17.5 | . | . | 28.0 | . | . | 28.1 | 20.8 | 17.7 | 30.8 |
| 2,4-DICHLOROPHENOL | 30.7 | 21.2 | . | 30.9 | 10.9 | . | . | 34.5 | 36.3 | 20.0 | . |
| 2,4,6-TRICHLOROPHENOL | 34.4 | 44.6 | . | 16.5 | 13.2 | . | . | 10.0 | 42.0 | 12.6 | 14.7 |
| 4-CHLORO-3-METHYLPHENOL | 28.8 | 27.4 | . | 20.6 | 19.2 | . | . | 24.6 | 29.2 | 11.3 | 18.8 |
| 2,4-DINITROPHENOL | 52.6 | 77.2 | . | . | 26.6 | . | . | 24.2 | 9.8 | 10.5 | 14.7 |
| 4,6-DINITRO-O-CRESOL | 56.7 | 42.5 | . | . | 21.6 | . | . | 44.7 | 8.8 | . | . |
| PENTACHLOROPHENOL | 19.2 | 39.0 | . | 44.4 | 28.0 | . | . | 28.3 | 19.3 | 14.5 | . |
| 4-NITROPHENOL | 12.5 | 32.5 | . | 50.0 | 19.9 | . | . | 16.4 | 29.9 | 24.0 | 23.5 |

TABLE I-8. RELATIVE STANDARD DEVIATIONS OF CONCENTRATIONS OF METALS AVERAGED OVER ALL SPIKE LEVELS (INCLUDING UNSPIKED SAMPLES)

| ELEMENT | POTW | IPW | CSB | PPS | COBS | EPS | EFBHD | IPW_IL | OSB_IL | IPL | OSBL |
|---|---|---|---|---|---|---|---|---|---|---|---|
| ANTIMONY | . | 15.3 | 43.1 | . | . | 5.2 | 6.5 | . | . | . | . |
| ARSENIC | . | 8.7 | 11.9 | 8.7 | . | 5.4 | 9.8 | . | . | . | . |
| BERYLLIUM | . | 3.0 | 14.4 | 6.2 | . | 5.4 | 0.0 | 6.2 | 6.2 | . | . |
| CADMIUM | . | 2.5 | 12.7 | 5.8 | . | 6.4 | 8.6 | 0.0 | 6.2 | 16.2 | 6.7 |
| CHROMIUM | . | 5.1 | 6.8 | 4.7 | . | 1.5 | 3.2 | 3.0 | 12.3 | 1.5 | 1.4 |
| COPPER | . | 6.2 | 5.6 | 2.4 | . | 2.5 | 1.5 | 4.7 | 7.9 | 1.7 | 0.7 |
| LEAD | . | 2.1 | 7.5 | 7.3 | . | 2.1 | 6.1 | 2.6 | 3.6 | 0.9 | 2.7 |
| MERCURY | . | 18.6 | 13.0 | 4.0 | . | 4.3 | 12.8 | . | . | . | . |
| NICKEL | . | 5.1 | 5.3 | 6.8 | . | 5.9 | 3.1 | 11.6 | 9.5 | 1.7 | 1.7 |
| SELENIUM | . | 7.7 | 20.4 | . | . | 11.3 | 10.2 | . | . | . | . |
| SILVER | . | 22.8 | 24.4 | 6.4 | . | . | 14.3 | 24.5 | 20.9 | . | . |
| THALLIUM | . | 11.4 | 19.8 | 10.1 | . | 15.9 | 3.1 | 5.3 | 6.7 | . | . |
| ZINC | . | 2.1 | 6.5 | 3.2 | . | 1.0 | 6.1 | 3.2 | 2.9 | 2.7 | 2.7 |

## C. Summary of the Average Mean Recoveries of Compounds in Spiked Residual Waste

## TABLE I-9. AVERAGE PERCENT RECOVERIES OF PURGEABLE ORGANICS IN SPIKED RESIDUAL WASTE SAMPLES

| COMPOUND | POTW | IPW | OSB | PPS | COBS | EPS | EFBHD | IPW_IL | OSB_IL | IPL | OSBL |
|---|---|---|---|---|---|---|---|---|---|---|---|
| METHYLENE CHLORIDE | 43.58 | . | . | 182.69 | 90.52 | . | . | . | 92.11 | 72.45 | 69.86 |
| TRICHLOROFLUORCMETHANE | . | . | . | . | . | . | . | . | 98.12 | . | . |
| 1,1-DICHLOROETHYLENE | 29.33 | . | . | 117.24 | 41.39 | . | . | 29.25 | 98.90 | 6.09 | . |
| 1,1-DICHLOROETHANE | 47.37 | . | . | 116.67 | 82.83 | . | . | 48.00 | 86.47 | 75.40 | . |
| TRANS-1,2-DICHLOROETHYLENE | 37.80 | . | . | 146.67 | 73.67 | . | . | 38.20 | 87.00 | 82.40 | 27.26 |
| CHLOROFORM | 50.91 | . | . | 114.17 | 88.00 | . | . | 55.03 | 95.81 | 82.72 | 32.20 |
| 1,2-DICHLOROETHANE | 58.00 | . | . | 252.50 | 110.83 | . | . | 15.10 | 86.82 | 95.52 | 81.22 |
| 1,1,1-TRICHLOROETHANE | 51.40 | . | . | 82.67 | 56.00 | . | . | 47.67 | 80.03 | 62.44 | . |
| CARBON TETRACHLORIDE | 52.40 | . | . | 73.67 | 43.75 | . | . | 45.75 | 88.14 | 53.84 | 10.30 |
| BROMODICHLOROMETHANE | 63.94 | . | . | 130.66 | 95.68 | . | . | 18.89 | 99.82 | . | . |
| 1,2-DICHLOROPROPANE | 54.20 | . | . | 198.33 | 84.33 | . | . | 14.25 | 113.79 | . | . |
| TRANS-1,3-DICHLORCPROPENE | 52.68 | . | . | 135.83 | 90.00 | . | . | 53.35 | 97.89 | . | . |
| TRICHLOROETHYLENE | 58.28 | . | . | . | 78.91 | . | . | 24.04 | 68.81 | 71.64 | . |
| DIBROMOCHLOROMETHANE | 62.00 | . | . | 0.00 | 107.17 | . | . | 41.37 | 96.98 | . | . |
| CIS-1,3-DICHLOROPROPENE | 52.59 | . | . | 0.00 | 95.38 | . | . | 55.98 | 91.98 | . | . |
| 1,1,2-TRICHLOROETHANE | 72.60 | . | . | 0.00 | 115.67 | . | . | 410.00 | 72.55 | 93.88 | 103.60 |
| BENZENE | 56.18 | . | . | 120.06 | 74.50 | . | . | 21.22 | 53.92 | 81.37 | 32.86 |
| BROMOFORM | 65.10 | . | . | 95.83 | 118.33 | . | . | 52.05 | 121.94 | . | . |
| 1,1,2,2-TETRACHLOROETHENE | 97.08 | . | . | 361.00 | 86.50 | . | . | 12.93 | 62.83 | 8.57 | . |
| 1,1,2,2-TETRACHLOROETHANE | 94.75 | . | . | 54.31 | 75.33 | . | . | 26.55 | 77.34 | 28.71 | . |
| TOLUENE | 90.94 | . | . | 76.67 | 78.17 | . | . | . | 97.25 | 37.30 | 16.86 |
| CHLOROBENZENE | 69.96 | . | . | 97.83 | 87.50 | . | . | 58.69 | 112.20 | 66.08 | 17.12 |
| ETHYLBENZENE | 77.28 | . | . | . | 86.83 | . | . | . | 100.73 | 72.30 | 11.40 |

## TABLE I-10. AVERAGE PERCENT RECOVERIES OF BASE/NEUTRAL EXTRACTABLE SEMIVOLATILE ORGANICS IN SPIKED RESIDUAL WASTE SAMPLES

| COMPOUND | POTW | IPW | OSB | PPS | COBS | EPS | EFBHD | IPW_IL | OSB_IL | IPL | OSBL |
|---|---|---|---|---|---|---|---|---|---|---|---|
| 1,3-DICHLOROBENZENE | 126.52 | 69.99 | . | 20.47 | 51.50 | . | . | 87.22 | 121.25 | . | . |
| 1,4-DICHLOROBENZENE | 72.40 | 72.75 | . | 15.98 | 62.51 | . | . | 51.45 | 48.46 | 66.88 | 66.45 |
| 1,2-DICHLOROBENZENE | 74.61 | 89.76 | . | 17.49 | 84.88 | . | . | 237.03 | 27.16 | 20.20 | 80.25 |
| HEXACHLOROETHANE | 124.13 | 82.13 | . | 55.30 | 69.00 | . | . | 178.78 | 34.04 | . | . |
| BIS(2-CHLOROETHYL) ETHER | 98.27 | 118.00 | . | 138.33 | 84.17 | . | . | 80.07 | 98.77 | 159.60 | 211.20 |
| BIS(2-CHLOROISOPROPYL) ETHER | 62.72 | 82.73 | . | 20.82 | 73.78 | . | . | 60.87 | 77.22 | 77.28 | 107.56 |
| HEXACHLOROBUTADIENE | 94.40 | 107.92 | . | 42.82 | 74.74 | . | . | 81.82 | 367.05 | 95.32 | 72.56 |
| NITROBENZENE | 23.98 | 86.79 | . | 92.41 | 74.83 | . | . | 28.49 | 261.21 | . | 147.00 |
| NAPHTHALENE | 74.33 | 82.31 | . | . | 75.46 | . | . | 53.58 | 54.84 | . | . |
| 1,2,4-TRICHLOROBENZENE | 142.89 | 133.18 | . | 19.70 | 24.17 | . | . | 127.73 | 109.13 | 80.40 | 206.80 |
| BIS(2-CHLOROETHOXY)METHANE | 52.75 | 70.33 | . | 43.41 | 84.19 | . | . | 71.59 | 191.52 | . | . |
| N-NITROSODI-N-PROPYLAMINE | 155.40 | 401.86 | . | 207.79 | 113.02 | . | . | 49.28 | 151.19 | 153.50 | 10.00 |
| HEXACHLOROCYCLOPENTADIENE | 8.22 | 29.70 | . | 17.32 | 22.92 | . | . | 18.30 | 158.68 | 91.20 | 70.80 |
| 2-CHLORONAPHTHALENE | 93.47 | 123.12 | . | 58.16 | 89.93 | . | . | 57.48 | 64.58 | . | . |
| ISOPHORONE | 31.69 | 63.90 | . | 34.50 | 74.81 | . | . | 117.87 | 66.00 | 89.04 | 102.24 |
| ACENAPHTHYLENE | 83.70 | 99.21 | . | 50.56 | 75.99 | . | . | 79.82 | 58.83 | 105.00 | 90.00 |
| ACENAPHTHENE | 80.08 | 95.43 | . | 46.08 | 75.91 | . | . | 235.74 | 40.11 | . | . |
| DIMETHYL PHTHALATE | 77.76 | 89.64 | . | 45.55 | 57.30 | . | . | 42.08 | 97.02 | 119.80 | 88.25 |
| 2,6-DINITROTOLUENE | 41.30 | 756.24 | . | 17.41 | 55.47 | . | . | 363.02 | 51.34 | 83.20 | 73.80 |
| FLUORENE | 77.44 | 96.84 | . | 52.94 | 76.66 | . | . | 81.67 | 89.58 | 80.07 | 101.60 |
| 2,4-DINITROTOLUENE | 32.79 | 78.54 | . | 19.04 | 59.33 | . | . | 70.62 | 56.02 | 78.36 | 69.56 |
| 1,2-DIPHENYLHYDRAZINE | 57.80 | 66.45 | . | 37.38 | 71.45 | . | . | 80.41 | 120.20 | . | . |
| 4-CHLOROPHENYL PHENYL ETHER | 86.57 | 122.64 | . | 48.79 | 76.00 | . | . | 73.74 | 63.06 | 79.60 | 187.80 |
| DIETHYL PHTHALATE | 81.28 | 169.86 | . | 33.22 | 95.33 | . | . | 64.09 | 131.74 | . | 165.96 |
| N-NITROSODIPHENYLAMINE | 283.32 | 655.30 | . | 36.67 | 41.00 | . | . | 135.56 | 85.40 | . | . |
| HEXACHLOROBENZENE | 82.71 | 98.57 | . | 43.82 | 78.86 | . | . | 88.48 | 2929.14 | 45.68 | 131.40 |
| 4-BROMOPHENYL PHENYL ETHER | 94.54 | 107.30 | . | 57.06 | 88.98 | . | . | 87.48 | 115.17 | 116.20 | 123.36 |
| PHENANTHRENE/ANTHRACENE | 72.37 | 84.52 | . | 38.22 | 66.28 | . | . | 10.84 | 73.24 | . | . |
| DI-N-BUTYL PHTHALATE | 83.41 | 79.66 | . | 32.17 | 96.02 | . | . | 57.30 | 83.93 | 75.90 | 95.20 |
| FLUORANTHENE | 107.56 | 74.67 | . | 52.76 | 92.75 | . | . | 63.55 | 101.00 | 85.96 | 111.72 |
| PYRENE | 115.58 | 81.37 | . | 54.31 | 101.86 | . | . | 53.15 | 100.18 | 309.20 | 100.00 |
| BENZIDINE | 33.43 | 33.80 | . | 17.42 | 31.69 | . | . | 56.63 | 39.03 | 10.00 | 15.60 |
| BUTYLBENZYL PHTHALATE | 196.85 | 157.66 | . | 32.60 | 67.18 | . | . | 93.27 | 68.34 | . | . |
| BIS(2-ETHYLHEXYL) PHTHALATE | 199.94 | 131.34 | . | . | 16.97 | . | . | 59.78 | 81.80 | . | . |
| CHRYSENE/BENZO(A)ANTHRACENE | 467.57 | 120.00 | . | 40.64 | 70.71 | . | . | 65.77 | 95.33 | 49.48 | 159.92 |
| 3,3'-DICHLOROBENZIDINE | 171.73 | 48.30 | . | 42.88 | 71.99 | . | . | 53.57 | 17.92 | 18.20 | 35.32 |
| DI-N-OCTYL PHTHALATE | 54.38 | 29.57 | . | 47.96 | 14.78 | . | . | 66.71 | . | 80.37 | 160.48 |
| BENZO(B)/BENZO(K)FLUORANTHENES | 206.82 | 159.92 | . | 65.28 | 118.47 | . | . | 58.30 | 70.37 | 55.60 | 193.25 |
| BENZO(A)PYRENE | 155.84 | 233.00 | . | 62.26 | 127.05 | . | . | 76.32 | 89.17 | . | . |
| INDENO(1,2,3-CD)PYRENE | 247.89 | 163.83 | . | . | . | . | . | . | . | . | . |
| DIBENZO(AH)ANTHRACENE | 228.45 | 193.97 | . | . | . | . | . | . | . | . | . |
| BENZO(GHI)PERYLENE | 221.74 | 180.65 | . | 48.94 | 106.29 | . | . | 123.73 | 150.06 | . | . |

TABLE I-11. AVERAGE PERCENT RECOVERIES OF ACID EXTRACTABLE SEMIVOLATILE ORGANICS IN SPIKED RESIDUAL WASTE SAMPLES

| COMPOUND | POTW | IPW | OSB | PPS | COBS | EPS | EFBHD | IPW_IL | OSB_IL | IPL | OSBL |
|---|---|---|---|---|---|---|---|---|---|---|---|
| 2-CHLOROPHENOL | 69.70 | 92.51 | . | 82.55 | 111.78 | . | . | 110.48 | 69.38 | 4.00 | 36.28 |
| 2-NITROPHENOL | 34.27 | 100.18 | . | 11.19 | 133.08 | . | . | 122.55 | 18.24 | . | . |
| PHENOL | 68.61 | 69.83 | . | 45.27 | 118.75 | . | . | 86.45 | 78.99 | 33.60 | 125.32 |
| 2,4-DIMETHYLPHENOL | 25.67 | 22.56 | . | 11.20 | 20.99 | . | . | 55.56 | 21.03 | 78.80 | 32.80 |
| 2,4-DICHLOROPHENOL | 60.13 | 46.16 | . | 48.10 | 125.77 | . | . | 136.24 | 74.03 | 78.00 | 25.00 |
| 2,4,6-TRICHLOROPHENOL | 104.42 | 123.11 | . | 60.41 | 119.52 | . | . | 115.20 | 86.03 | 117.00 | 19.96 |
| 4-CHLORO-3-METHYLPHENOL | 61.47 | 102.50 | . | 19.71 | 108.75 | . | . | 117.70 | 57.33 | . | . |
| 2,4-DINITROPHENOL | 45.20 | 92.30 | . | 11.25 | 116.50 | . | . | 109.17 | 18.25 | 97.60 | 41.48 |
| 4,6-DINITRO-O-CRESOL | 52.61 | 31.48 | . | 11.20 | 101.45 | . | . | 122.20 | 18.08 | . | . |
| PENTACHLOROPHENOL | 139.33 | 131.99 | . | 59.84 | 90.18 | . | . | 152.31 | 60.38 | 102.50 | 0.00 |
| 4-NITROPHENOL | 663.25 | 103.85 | . | 17.46 | 118.14 | . | . | 103.23 | 21.91 | 105.20 | 46.20 |

## TABLE I-12. AVERAGE PERCENT RECOVERIES OF METALS IN SPIKED RESIDUAL WASTE SAMPLES

| ELEMENT | POTW | IPW | GSB | PPS | COBS | EPS | EFBHD | IPW_IL | OSB_IL | IPL | OSBL |
|---|---|---|---|---|---|---|---|---|---|---|---|
| ANTIMONY | . | 80.98 | 1.68 | 5.56 | . | 53.78 | 74.40 | . | . | . | . |
| ARSENIC | . | 103.93 | 91.68 | 4.44 | . | 69.44 | 26.67 | . | . | . | . |
| BERYLLIUM | . | 101.94 | 83.72 | 72.33 | . | 93.60 | 86.40 | 121.43 | 83.53 | . | . |
| CADMIUM | . | 95.76 | 92.92 | 103.33 | . | 96.22 | 65.33 | 98.57 | 103.53 | 106.49 | 103.86 |
| CHROMIUM | . | 109.78 | 99.00 | 106.67 | . | 110.09 | 114.67 | 108.86 | 132.35 | 94.12 | 48.21 |
| COPPER | . | 94.53 | 86.84 | 98.78 | . | . | 94.40 | 96.55 | 89.41 | 100.12 | 99.36 |
| LEAD | . | 91.54 | 98.60 | 98.89 | . | 97.33 | 88.67 | 105.69 | 120.00 | 102.86 | 107.94 |
| MERCURY | . | 68.85 | 74.40 | . | . | 81.68 | 112.53 | . | . | . | . |
| NICKEL | . | 100.00 | 97.50 | 69.11 | . | 87.35 | 98.53 | 99.00 | 82.59 | 106.08 | 99.01 |
| SELENIUM | . | 97.78 | [illegible] | 0.00 | . | 53.58 | 60.00 | . | . | . | . |
| SILVER | . | 2.30 | 72.60 | 23.33 | . | 0.00 | . | 3.86 | 22.12 | . | . |
| THALLIUM | . | 75.41 | 42.80 | 72.22 | . | 86.67 | 73.67 | 98.06 | 94.82 | . | . |
| ZINC | . | 102.61 | 93.50 | 117.22 | . | 110.54 | . | 126.90 | 95.29 | 114.46 | 114.22 |

# Appendix II. Potential Mobility (Leachate) Interlaboratory Study Data

## Legend for Verification Analyses and Quality Assurance for Potential Mobility (Leachate) Data*

| Symbols | Identification |
|---|---|
| . | Not determined. |
| < | Lower detection limit of method. |
| ( ) | Found to be a statistical outlier and not included in the calculations. |
| Z | Standard deviation was not calculated because all points in the data set were less than lower detection limit of method and/or zero. |
| N | Percent recovery was calculated to be less than zero. |

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* Concentrations are reported as microgram of compound per liter of leaching medium (parts per billion).

## A. Ink Pigment Leachate (Interlaboratory)

## TABLE II-1. PURGEABLE ORGANICS DATA— INK PIGMENT LEACHATE, SPIKE LEVEL 0

| CUMPOUND | ADDED | NO_1 | NO_2 | NO_3 | NO_4 | NO_5 | MEAN | S | RSD | RECOVERY |
|---|---|---|---|---|---|---|---|---|---|---|
| METHYLENE CHLORIDE | 0 | 800 | 1680 | 1036 | 1320 | 1170 | 1201.2 | 328.86 | 27.4 | . |
| 1,1-DICHLOROETHYLENE | 0 | 59 | 74 | 74 | 79 | 85 | 74.2 | 9.63 | 13.0 | . |
| 1,1-DICHLOROETHANE | 0 | 18 | 27 | 19 | 32 | 15 | 22.2 | 7.05 | 31.8 | . |
| TRANS-1,2-DICHLOROETHYLENE | 0 | 26 | 22 | 26 | 28 | 28 | 26 | 2.45 | 9.4 | . |
| CHLOROFORM | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| 1,2-DICHLOROETHANE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| 1,1,1-TRICHLOROETHANE | 0 | 26 | 38 | 26 | 34 | 38 | 32.4 | 6.07 | 18.7 | . |
| CARBON TETRACHLORIDE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| BROMODICHLOROMETHANE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| 1,2-DICHLOROPROPANE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| TRANS-1,3-DICHLOROPROPENE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| TRICHLOROETHYLENE | 0 | 597 | 802 | 745 | 888 | 773 | 761 | 106.19 | 14.0 | . |
| DIBROMOCHLOROMETHANE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| CIS-1,3-DICHLOROPROPENE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| 1,1,2-TRICHLOROETHANE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| BENZENE | 0 | 318 | 400 | 460 | 481 | 500 | 431.8 | 73.87 | 17.1 | . |
| BROMOFORM | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| 1,1,2,2-TETRACHLOROETHENE | 0 | 182 | (1550) | 277 | 407 | 241 | 276.75 | 95.26 | 34.4 | . |
| 1,1,2,2-TETRACHLOROETHANE | 0 | 121 | (959) | 152 | 261 | 139 | 168.25 | 63.13 | 37.5 | . |
| TOLUENE | 0 | (1920) | 3220 | 3570 | 3580 | 3730 | 3525 | 216.10 | 6.1 | . |
| CHLOROBENZENE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| ETHYLBENZENE | 0 | 930 | 1140 | 2090 | 2230 | 2210 | 1720 | 631.98 | 36.7 | . |

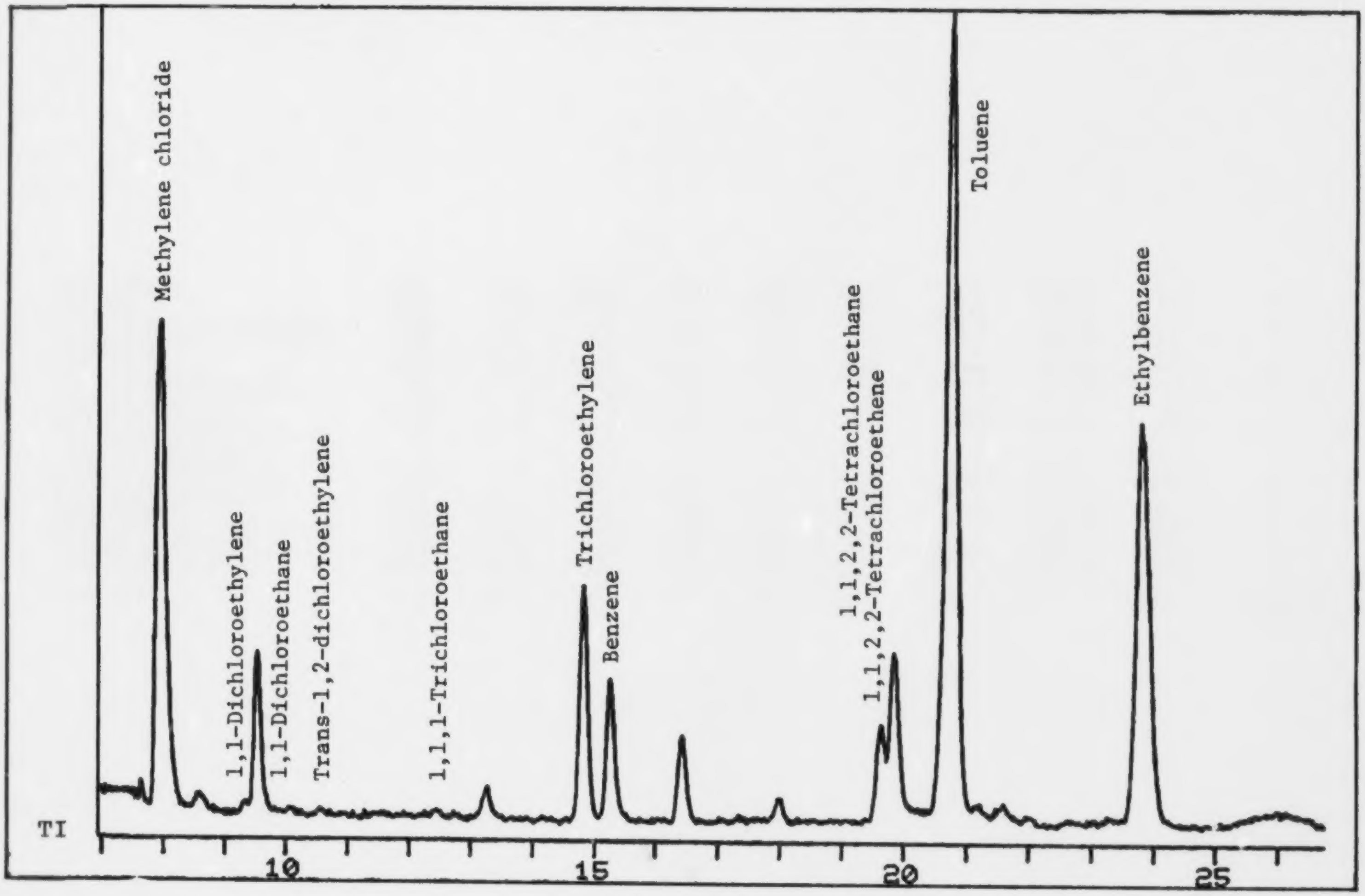

Figure II-1. GC/MS chromatogram of purgeable organics by purge and trap—Ink Pigment Leachate, unspiked

## TABLE II-2. PURGEABLE ORGANICS DATA— INK PIGMENT LEACHATE, SPIKE LEVEL I

| COMPOUND | ADDED | NO_1 | NO_2 | NO_3 | NO_4 | NO_5 | MEAN | S | RSD | RECOVERY |
|---|---|---|---|---|---|---|---|---|---|---|
| METHYLENE CHLORIDE | 2500 | 2780 | (5660) | 2900 | 3380 | 2990 | 3012.5 | 259.66 | 8.6 | 72 |
| 1,1-DICHLOROETHYLENE | 500 | 114 | 101 | 88.3 | 111 | 109 | 104.66 | 10.34 | 9.9 | 6 |
| 1,1-DICHLOROETHANE | 500 | 394 | 320 | 408 | 450 | 424 | 399.2 | 48.92 | 12.3 | 75 |
| TRANS-1,2-DICHLOROETHYLENE | 2500 | 1990 | 1940 | 2110 | 2200 | 2190 | 2086 | 117.18 | 5.6 | 82 |
| CHLOROFORM | 500 | 381 | 383 | 401 | 466 | 437 | 413.6 | 36.92 | 8.9 | 83 |
| 1,2-DICHLOROETHANE | 2500 | 2450 | 2330 | 2390 | 2440 | 2330 | 2388 | 57.62 | 2.4 | 96 |
| 1,1,1-TRICHLOROETHANE | 500 | 334 | 256 | 321 | 416 | 396 | 344.6 | 63.76 | 18.5 | 62 |
| CARBON TETRACHLORIDE | 2500 | 1380 | 840 | 1170 | 1740 | 1600 | 1346 | 356.34 | 26.5 | 54 |
| BROMODICHLOROMETHANE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| 1,2-DICHLOROPROPANE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| TRANS-1,3-DICHLOROPROPENE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| TRICHLOROETHYLENE | 2500 | 2490 | 2570 | 2380 | 2830 | 2490 | 2552 | 169.47 | 6.6 | 72 |
| DIBROMOCHLOROMETHANE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| CIS-1,3-DICHLOROPROPENE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| 1,1,2-TRICHLOROETHANE | 500 | 467 | 382 | 493 | 490 | 515 | 469.4 | 51.73 | 11.0 | 94 |
| BENZENE | 2500 | 2380 | 2390 | 2440 | 2730 | 2390 | 2466 | 149.43 | 6.1 | 81 |
| BROMOFORM | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| 1,1,2,2-TETRACHLOROETHENE | 500 | 379 | 226 | 253 | 421 | 319 | 319.6 | 82.17 | 25.7 | 9 |
| 1,1,2,2-TETRACHLOROETHANE | 500 | 366 | 248 | 252 | 403 | 290 | 311.8 | 69.60 | 22.3 | 29 |
| TOLUENE | 2500 | 4330 | (6490) | 4350 | 4830 | 4320 | 4457.5 | 248.65 | 5.6 | 37 |
| CHLOROBENZENE | 2500 | 1760 | 1160 | 1520 | 2130 | 1690 | 1652 | 353.94 | 21.4 | 66 |
| ETHYLBENZENE | 2500 | 3790 | (5420) | 3180 | 3760 | 3380 | 3527.5 | 297.48 | 8.4 | 72 |

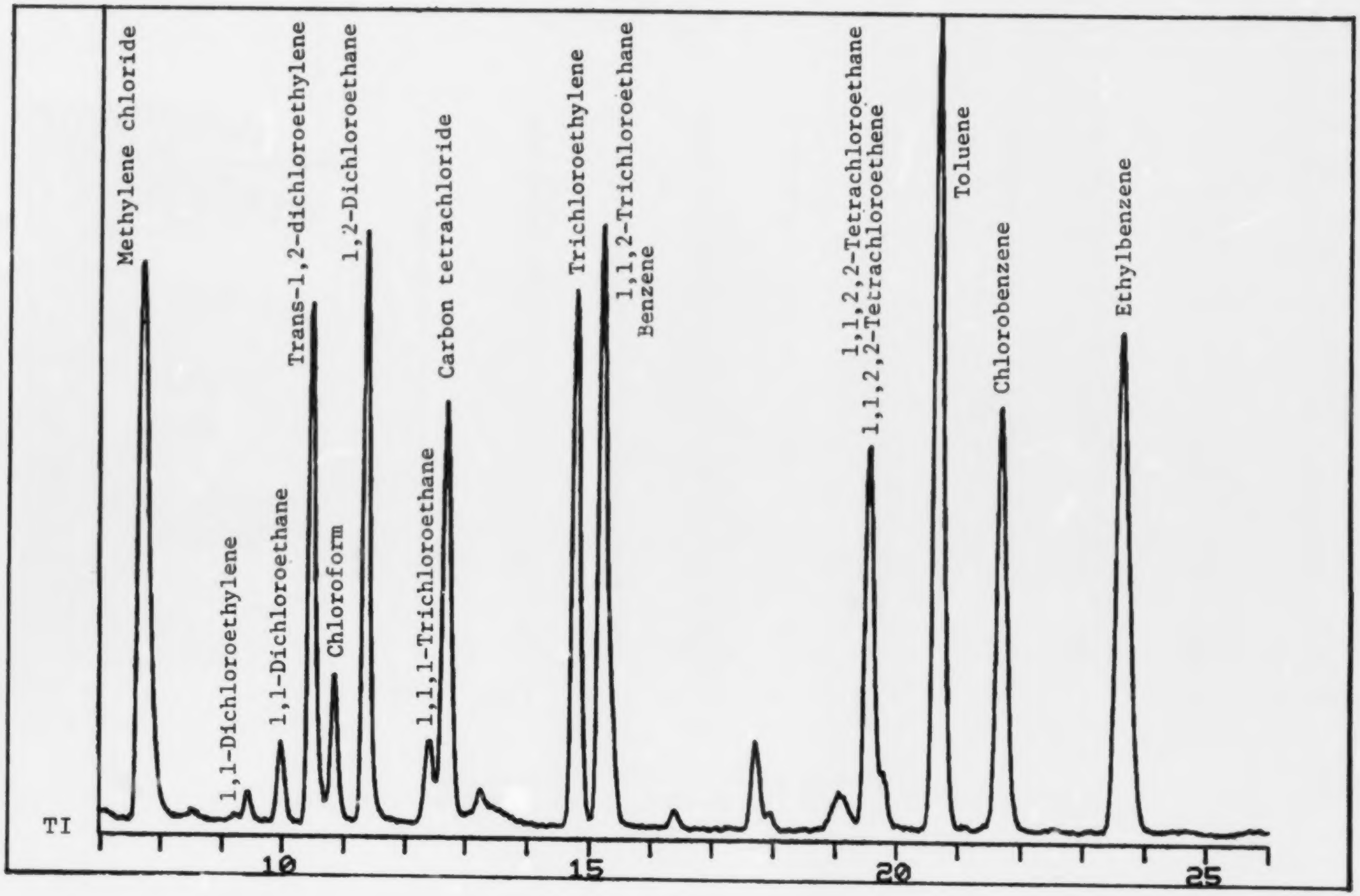

Figure II-2. GC/MS chromatogram of purgeable organics by purge and trap—Ink Pigment Leachate, spiked

### TABLE II-3. BASE/NEUTRAL EXTRACTABLE SEMIVOLATILE ORGANICS DATA— INK PIGMENT LEACHATE, SPIKE LEVEL 0

| COMPOUND | ADDED | NO_1 | NO_2 | NO_3 | NO_4 | NO_5 | MEAN | S | RSD | RECOVERY |
|---|---|---|---|---|---|---|---|---|---|---|
| 1,3-DICHLOROBENZENE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| 1,4-DICHLOROBENZENE | 0 | 34 | 95 | 105 | 78 | 84 | 79.2 | 27.31 | 34.5 | . |
| 1,2-DICHLOROBENZENE | 0 | 128 | 287 | 246 | 261 | 335 | 251.4 | 76.82 | 30.6 | . |
| HEXACHLOROETHANE | 0 | 19 | 41 | 31 | 49 | 65 | 41 | 17.49 | 42.7 | . |
| BIS(2-CHLOROETHYL) ETHER | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| BIS(2-CHLOROISOPROPYL) ETHER | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| HEXACHLOROBUTADIENE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| NITROBENZENE | 0 | 171 | 222 | 281 | 259 | 245 | 235.6 | 42.00 | 17.8 | . |
| NAPHTHALENE | 0 | 306 | 371 | 568 | 762 | 804 | 562.2 | 223.95 | 39.8 | . |
| 1,2,4-TRICHLOROBENZENE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| BIS(2-CHLOROETHOXY)METHANE | 0 | 207 | 194 | 241 | 245 | 258 | 229 | 27.16 | 11.9 | . |
| N-NITROSODI-N-PROPYLAMINE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| HEXACHLOROCYCLOPENTADIENE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| 2-CHLORONAPHTHALENE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| ISOPHORONE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| ACENAPHTHYLENE | 0 | 28 | 45 | 48 | 47 | 55 | 44.6 | 10.01 | 22.5 | . |
| ACENAPHTHENE | 0 | <10 | 25 | 16 | 13 | 14 | <15.6 | 5.68 | 36.4 | . |
| DIMETHYL PHTHALATE | 0 | 17 | 48 | 59 | 29 | 19 | 34.4 | 18.43 | 53.6 | . |
| 2,6-DINITROTOLUENE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| FLUORENE | 0 | <10 | (16) | (29) | 11 | 11 | <10.6667 | 0.58 | 5.4 | . |
| 2,4-DINITROTOLUENE | 0 | 0 | 72 | 113 | 59 | 39 | 56.6 | 41.64 | 73.6 | . |
| 1,2-DIPHENYLHYDRAZINE | 0 | 21 | 59 | 81 | 30 | 18 | 41.8 | 27.25 | 65.2 | . |
| 4-CHLOROPHENYL PHENYL ETHER | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| DIETHYL PHTHALATE | 0 | 433 | 715 | 1150 | 453 | 363 | 622.8 | 323.57 | 52.0 | . |
| N-NITROSODIPHENYLAMINE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| HEXACHLOROBENZENE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| 4-BROMOPHENYL PHENYL ETHER | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| PHENANTHRENE/ANTHRACENE | 0 | <10 | <10 | 11 | <10 | <10 | <10.2 | 0.45 | 4.4 | . |
| DI-N-BUTYL PHTHALATE | 0 | -10 | 12 | (21) | <10 | <10 | <10.5 | 1.00 | 9.5 | . |
| FLUORANTHENE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| PYRENE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| BENZIDINE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| BUTYLBENZYL PHTHALATE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| BIS(2-ETHYLHEXYL) PHTHALATE | 0 | <10 | 741 | 46 | 1090 | <10 | <379.4 | 504.92 | 133.1 | . |
| CHRYSENE/BENZO(A)ANTHRACENE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| 3,3'-DICHLOROBENZIDINE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| DI-N-OCTYL PHTHALATE | 0 | 17 | 78 | 11 | 156 | <10 | <54.4 | 63.51 | 116.7 | . |
| BENZO(B)/BENZO(K)FLUORANTHENES | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| BENZO(A)PYRENE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| INDENO(1,2,3-CD)PYRENE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| DIBENZO(AH)ANTHRACENE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| BENZO(GHI)PERYLENE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |

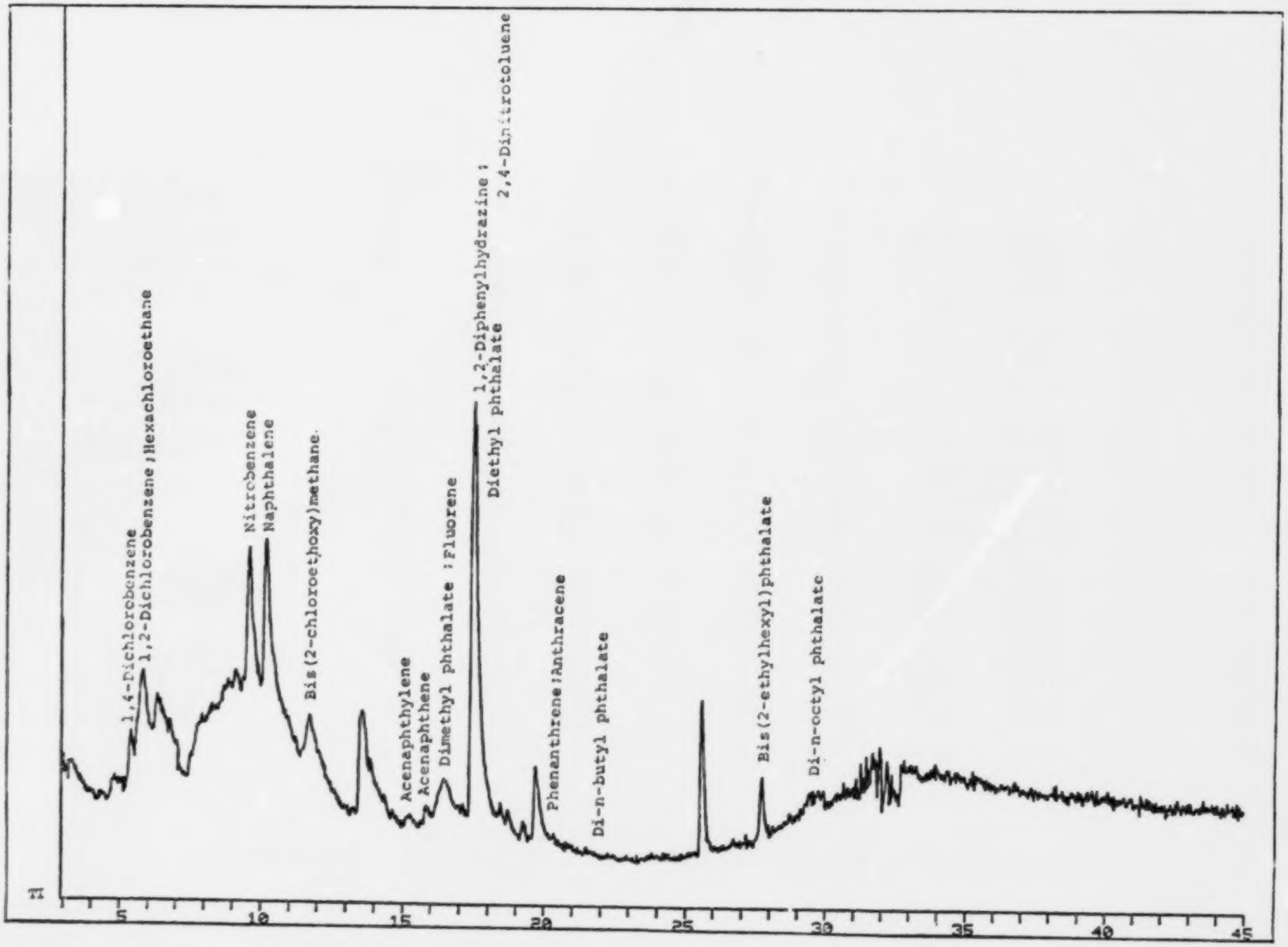

Figure II-3. GC/MS chromatogram of base/neutral extractables—Ink Pigment Leachate, unspiked

TABLE II-4. BASE/NEUTRAL EXTRACTABLE SEMIVOLATILE ORGANICS DATA— INK PIGMENT LEACHATE, SPIKE LEVEL I

| COMPOUND | ADDED | NO_1 | NO_2 | NO_3 | NO_4 | NO_5 | MEAN | S | RSD | RECOVERY |
|---|---|---|---|---|---|---|---|---|---|---|
| 1,3-DICHLOROBENZENE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| 1,4-DICHLOROBENZENE | 500 | 442 | 331 | 461 | 364 | 470 | 413.6 | 62.28 | 15.1 | 67 |
| 1,2-DICHLOROBENZENE | 100 | 303 | 248 | 306 | 232 | 269 | 271.6 | 32.79 | 12.1 | 20 |
| HEXACHLOROETHANE | 0 | 31 | 25 | 31 | 23 | 23 | 26.6 | 4.10 | 15.4 | . |
| BIS(2-CHLOROETHYL) ETHER | 100 | 167 | 140 | 155 | 138 | 198 | 159.6 | 24.50 | 15.4 | 160 |
| BIS(2-CHLOROISOPROPYL) ETHER | 500 | 413 | 358 | 402 | 322 | 437 | 386.4 | 46.00 | 11.9 | 77 |
| HEXACHLOROBUTADIENE | 500 | 447 | 476 | 399 | 486 | 575 | 476.6 | 64.55 | 13.5 | 95 |
| NITROBENZENE | 100 | 70 | 52 | 55 | 77 | 91 | 69 | 16.08 | 23.3 | N |
| NAPHTHALENE | 0 | 661 | 588 | 430 | 333 | 387 | 479.8 | 138.91 | 29.0 | . |
| 1,2,4-TRICHLOROBENZENE | 100 | 85 | 88 | 63 | 77 | 89 | 80.4 | 10.81 | 13.4 | 80 |
| BIS(2-CHLOROETHOXY)METHANE | 0 | 165 | 159 | 182 | 153 | 203 | 172.4 | 20.24 | 11.7 | . |
| N-NITROSODI-N-PROPYLAMINE | 100 | 178 | 138 | (58) | 137 | 161 | 153.5 | 19.74 | 12.9 | 153 |
| HEXACHLOROCYCLOPENTADIENE | 100 | 91 | 93 | 94 | 81 | 97 | 91.2 | 6.10 | 6.7 | 91 |
| 2-CHLORONAPHTHALENE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| ISOPHORONE | 500 | 425 | 377 | 433 | 440 | 551 | 445.2 | 64.09 | 14.4 | 89 |
| ACENAPHTHYLENE | 100 | 172 | 152 | 169 | 113 | 142 | 149.6 | 23.88 | 16.0 | 105 |
| ACENAPHTHENE | 0 | 10 | 12 | 13 | <10 | <10 | <11 | 1.41 | 12.9 | . |
| DIMETHYL PHTHALATE | 100 | 162 | 151 | 159 | 124 | 175 | 154.2 | 18.97 | 12.3 | 120 |
| 2,6-DINITROTOLUENE | 100 | 95 | 77 | 86 | 91 | 67 | 83.2 | 11.28 | 13.6 | 83 |
| FLUORENE | 500 | 425 | 404 | 410 | 286 | 530 | 411 | 86.65 | 21.1 | 80 |
| 2,4-DINITROTOLUENE | 500 | 420 | 355 | 440 | 416 | 611 | 448.4 | 96.29 | 21.5 | 78 |
| 1,2-DIPHENYLHYDRAZINE | 0 | <10 | <10 | <10 | 10 | 13 | <10.6 | 1.34 | 12.7 | . |
| 4-CHLOROPHENYL PHENYL ETHER | 100 | 83 | 76 | 86 | 50 | 103 | 79.6 | 19.30 | 24.2 | 80 |
| DIETHYL PHTHALATE | 500 | 600 | 564 | 599 | 479 | 631 | 574.6 | 58.47 | 10.2 | N |
| N-NITROSODIPHENYLAMINE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| HEXACHLOROBENZENE | 500 | 241 | 229 | 237 | 213 | 222 | 228.4 | 11.30 | 4.9 | 46 |
| 4-BROMOPHENYL PHENYL ETHER | 500 | 610 | 585 | 624 | 525 | 561 | 581 | 39.50 | 6.8 | 116 |
| PHENANTHRENE/ANTHRACENE | 0 | <10 | <10 | <10 | <10 | <10 | <10 | Z | . | . |
| DI-N-BUTYL PHTHALATE | 100 | 90 | 87 | 95 | 76 | 84 | 86.4 | 7.09 | 8.2 | 76 |
| FLUORANTHENE | 500 | 421 | 435 | 502 | 395 | 396 | 429.8 | 43.79 | 10.2 | 86 |
| PYRENE | 100 | 315 | 283 | 320 | 267 | 361 | 309.2 | 36.40 | 11.8 | 309 |
| BENZIDINE | 100 | <10 | <10 | <10 | <10 | <10 | <10 | Z | . | 10 |
| BUTYLBENZYL PHTHALATE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| BIS(2-ETHYLHEXYL) PHTHALATE | 0 | <10 | <10 | (275) | <10 | 65 | <23.75 | 27.50 | 115.8 | . |
| CHRYSENE/BENZO(A)ANTHRACENE | 500 | 246 | 251 | 273 | 227 | 240 | 247.4 | 16.89 | 6.8 | 49 |
| 3,3'-DICHLOROBENZIDINE | 500 | 88 | 76 | 98 | 94 | 99 | 91 | 9.43 | 10.4 | 18 |
| DI-N-OCTYL PHTHALATE | 500 | 436 | 459 | (765) | 440 | 490 | 456.25 | 24.64 | 5.4 | 80 |
| BENZO(B)/BENZO(K)FLUORANTHENES | 100 | 47 | 58 | 54 | 58 | 61 | 55.6 | 5.41 | 9.7 | 56 |
| BENZO(A)PYRENE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| INDENO(1,2,3-CD)PYRENE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| DIBENZO(AH)ANTHRACENE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| BENZO(GHI)PERYLENE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |

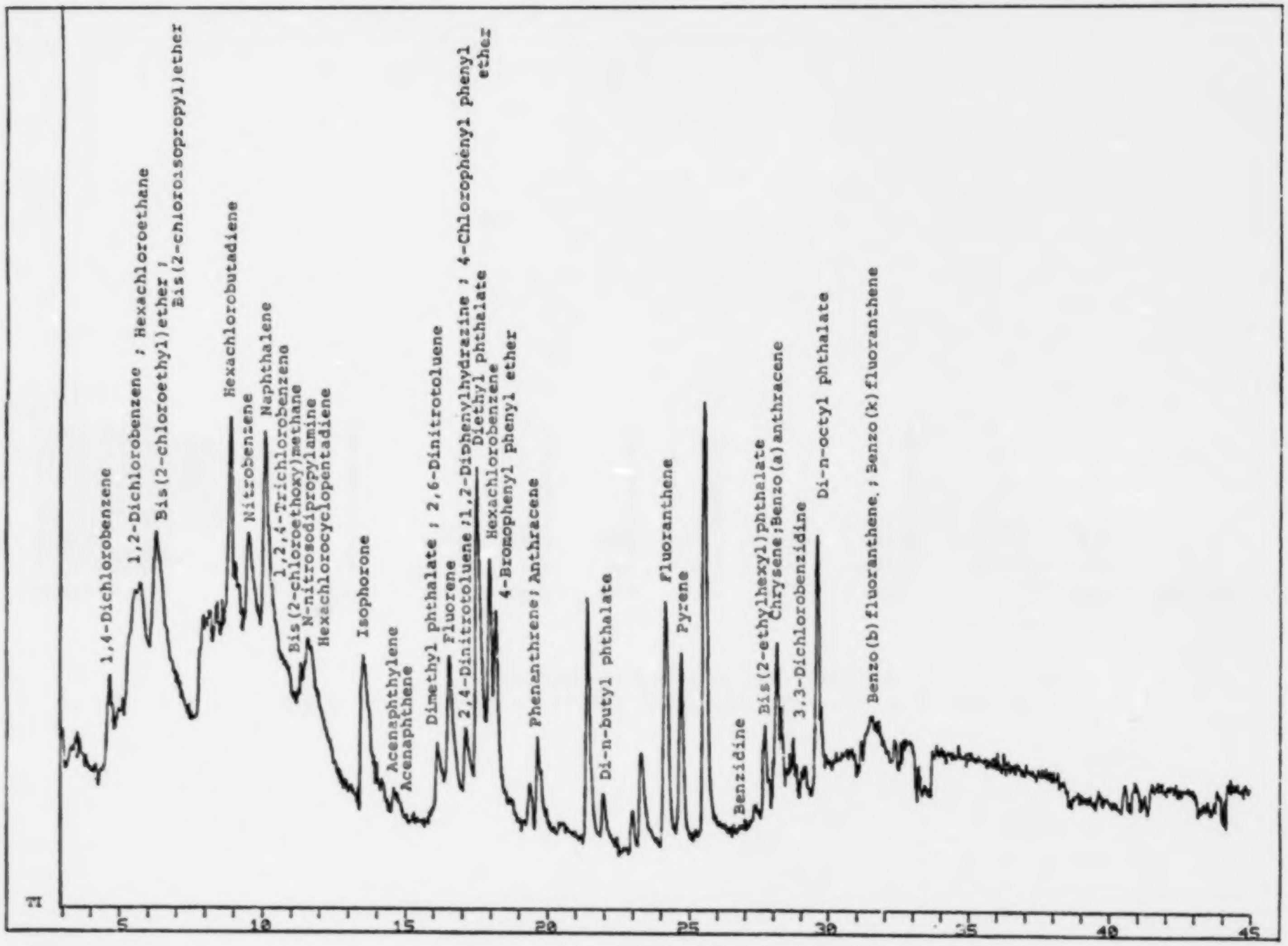

Figure II-4. GC/MS chromatogram of base/neutral extractables—
Ink Pigment Leachate, spiked

## TABLE II-5. ACID EXTRACTABLE SEMIVOLATILE ORGANICS DATA—INK PIGMENT LEACHATE, SPIKE LEVEL 0

| COMPOUND | ADDED | NO_1 | NO_2 | NO_3 | NO_4 | NO_5 | MEAN | S | RSD | RECOVERY |
|---|---|---|---|---|---|---|---|---|---|---|
| 2-CHLOROPHENOL | 0 | (218) | 439 | 495 | 416 | 421 | 442.75 | 36.21 | 8.2 | . |
| 2-NITROPHENOL | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| PHENOL | 0 | 3060 | 3330 | 3100 | 3210 | 2840 | 3108 | 182.95 | 5.9 | . |
| 2,4-DIMETHYLPHENOL | 0 | 89 | 126 | 95 | 104 | 92 | 101.2 | 14.96 | 14.8 | . |
| 2,4-DICHLOROPHENOL | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| 2,4,6-TRICHLOROPHENOL | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| 4-CHLORO-3-METHYLPHENOL | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| 2,4-DINITROPHENOL | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| 4,6-DINITRO-O-CRESOL | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| PENTACHLOROPHENOL | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| 4-NITROPHENOL | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |

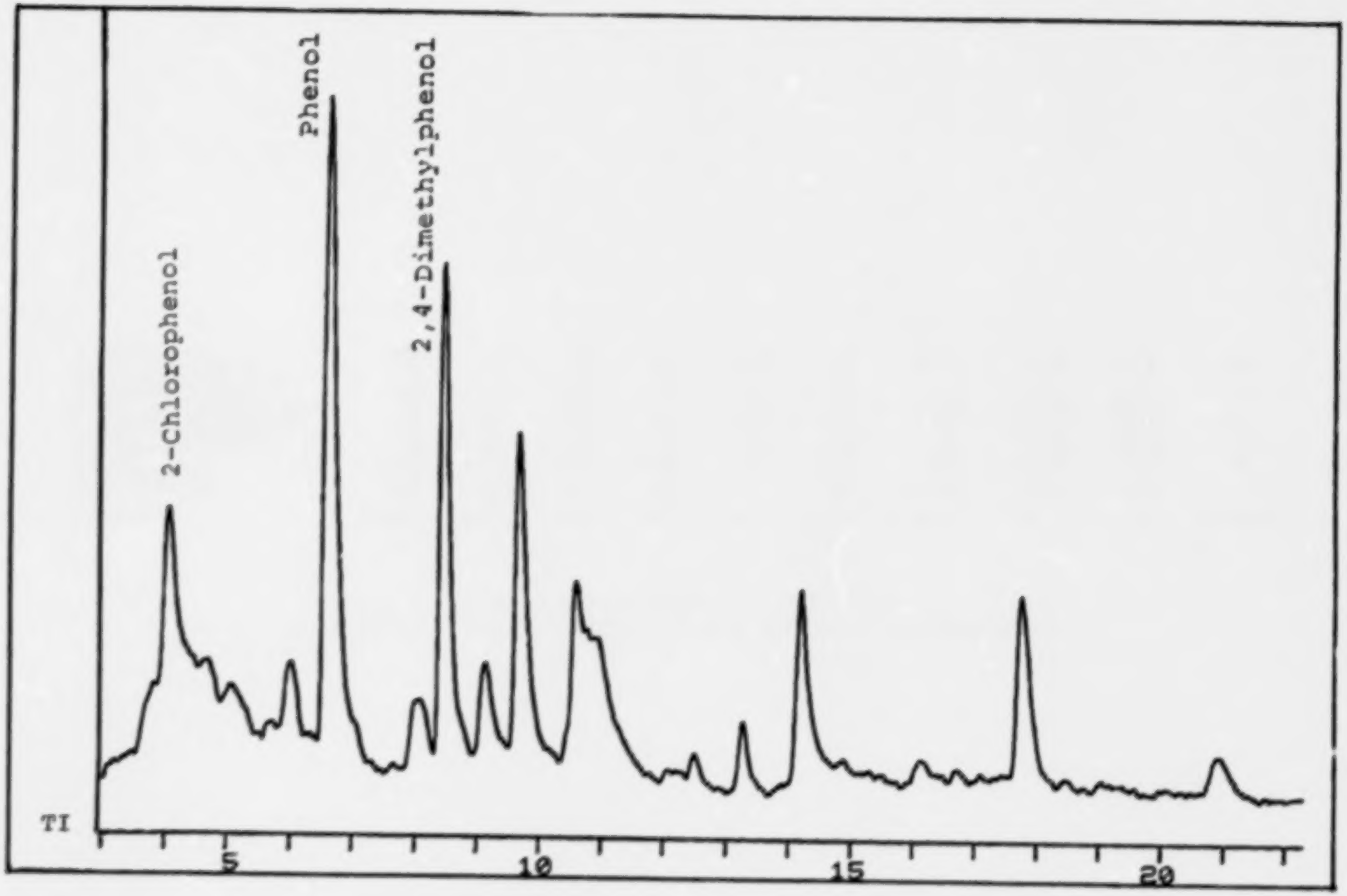

Figure II-5. GC/MS chromatogram of acid extractables—
Ink Pigment Leachate, unspiked

## TABLE II-6. ACID EXTRACTABLE SEMIVOLATILE ORGANICS DATA—INK PIGMENT LEACHATE, SPIKE LEVEL I

| COMPOUND | ADDED | NO_1 | NO_2 | NO_3 | NO_4 | NO_5 | MEAN | S | RSD | RECOVERY |
|---|---|---|---|---|---|---|---|---|---|---|
| 2-CHLOROPHENOL | 500 | (795) | 475 | 510 | 460 | 406 | 462.75 | 43.25 | 9.3 | [illegible] |
| 2-NITROPHENOL | 0 | 0 | 0 | 0 | 0 | 3 | 0 | [illegible] | [illegible] | [illegible] |
| PHENOL | 1000 | 4210 | 3350 | 3090 | 3270 | 3300 | 3444 | [illegible] | [illegible] | [illegible] |
| 2,4-DIMETHYLPHENOL | 100 | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] |
| 2,4-DICHLOROPHENOL | 100 | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] |
| 2,4,6-TRICHLOROPHENOL | 100 | [illegible] | 75 | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] |
| 4-CHLORO-3-METHYLPHENOL | 500 | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] |
| 2,4-DINITROPHENOL | 0 | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] |
| 4,6-DINITRO-O-CRESOL | 500 | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | 10.5 | [illegible] |
| PENTACHLOROPHENOL | 100 | (<25) | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] |
| 4-NITROPHENOL | 100 | 74 | 105 | 141 | 115 | 91 | 105.2 | [illegible] | [illegible] | 105 |

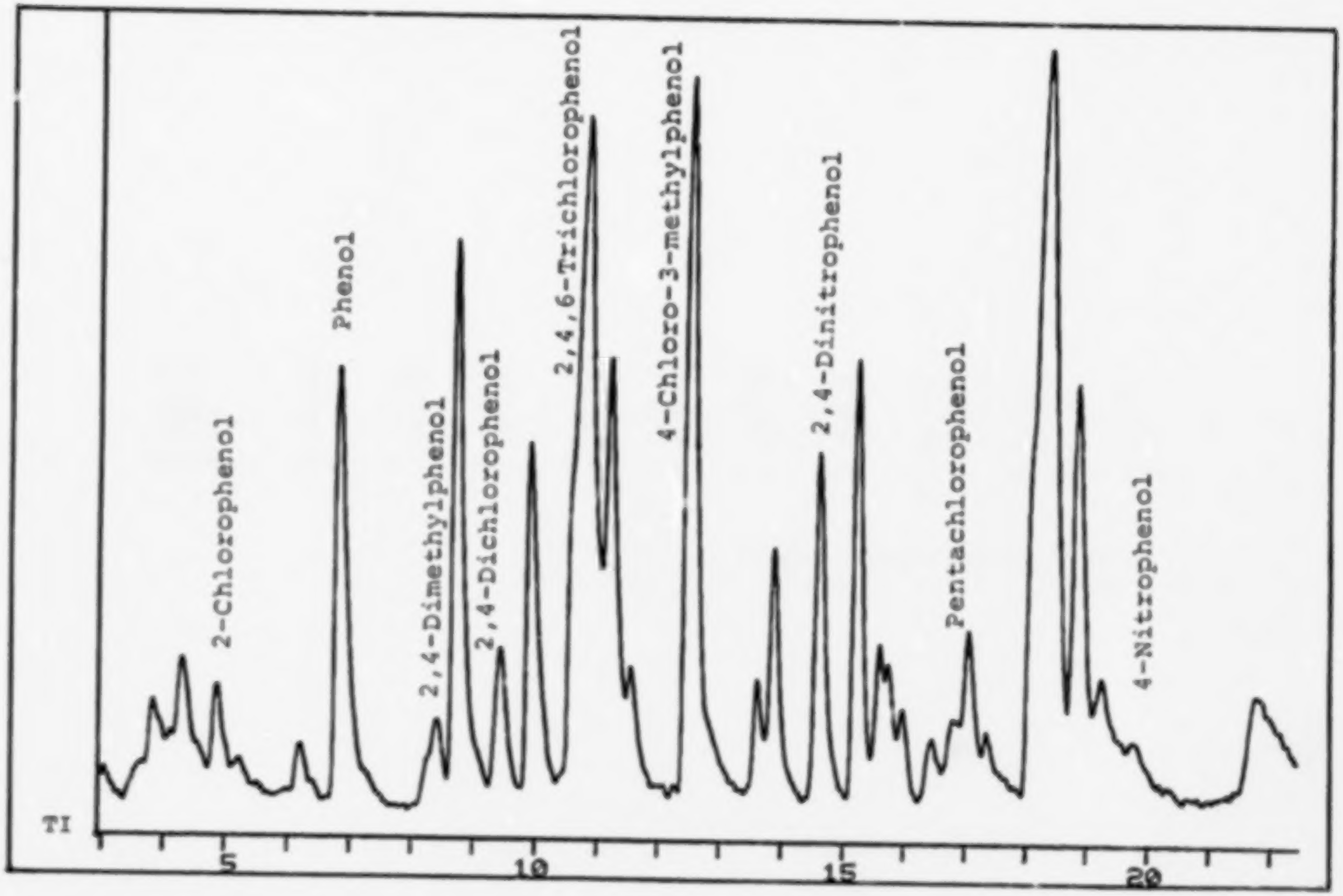

Figure II-6. GC/MS chromatogram of acid extractables—
Ink Pigment Leachate, spiked

## TABLE II-7. METALS DATA— INK PIGMENT LEACHATE, SPIKE LEVEL 0

| ELEMENT | ADDED | NO_1 | NO_2 | NO_3 | NO_4 | NO_5 | MEAN | S | RSD | RECOVERY |
|---|---|---|---|---|---|---|---|---|---|---|
| CADMIUM | 0 | 12 | 8 | 8 | 4 | 4 | 7.2 | 3.35 | 46.5 | * |
| CHROMIUM | 0 | [illegible] | [illegible] | [illegible] | [illegible] | 343 | [illegible] | 6.26 | 1.8 | * |
| COPPER | 0 | <50 | <50 | <50 | <50 | <50 | <50 | . | . | * |
| LEAD | 0 | 7610 | 7460 | 7510 | 7510 | 7610 | 7540 | 67.08 | 0.9 | * |
| NICKEL | 0 | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | 0.00 | 0.0 | * |
| ZINC | 0 | 1560 | 1480 | 1560 | 1650 | 1560 | 1562 | 60.17 | 3.9 | * |

### TABLE II-8. METALS DATA—
### INK PIGMENT LEACHATE, SPIKE LEVEL I

| ELEMENT | ADDED | NO_1 | NO_2 | NO_3 | NO_4 | NO_5 | MEAN | S | RSD | RECOVERY |
|---|---|---|---|---|---|---|---|---|---|---|
| CADMIUM | 200 | 213 | 221 | 212 | 212 | 217 | [illegible] | [illegible] | 1.7 | 108 |
| CHROMIUM | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] |
| COPPER | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] |
| LEAD | 10000 | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] |
| NICKEL | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] |
| ZINC | 2000 | 3700 | 3703 | 3700 | 4050 | 3880 | 3842 | [illegible] | [illegible] | 114 |

# TABLE II-9. METALS DATA—
INK PIGMENT LEACHATE, SPIKE LEVEL II

| ELEMENT | ADDED | NU_1 | NO_2 | NO_3 | NO_4 | NO_5 | MEAN | S | RSD | RECOVERY |
|---|---|---|---|---|---|---|---|---|---|---|
| CADMIUM | 1000 | 1100 | 1090 | 1090 | 1100 | 1100 | 1096 | 5.48 | 0.5 | 109 |
| CHROMIUM | 5000 | 2150 | 2130 | 2160 | 2150 | 2150 | 2148 | 10.95 | 0.5 | 36 |
| COPPER | 1000 | 1040 | 1050 | 1090 | 1070 | 1070 | 1064 | 19.49 | 1.8 | 101 |
| LEAD | 50000 | 56500 | 56500 | 56500 | 57500 | 57500 | 56900 | 547.72 | 1.0 | 99 |
| NICKEL | 1000 | 1190 | 1170 | 1120 | 1120 | 1120 | 1144 | 33.62 | 2.9 | 108 |
| ZINC | 15000 | 18700 | 18700 | 18900 | 19000 | 18700 | 18800 | 141.42 | 0.8 | 115 |

## B. Organic Still Bottoms Leachate (Interlaboratory)

## TABLE II-10. PURGEABLE ORGANICS DATA— ORGANIC STILL BOTTOMS LEACHATE, SPIKE LEVEL 0

| COMPOUND | ADDED | NO_1 | NO_2 | NO_3 | NO_4 | NO_5 | MEAN | S | RSD | RECOVERY |
|---|---|---|---|---|---|---|---|---|---|---|
| METHYLENE CHLORIDE | 0 | (1080) | 738 | 776 | 706 | 815 | 758.75 | 47.17 | 6.2 | . |
| 1,1-DICHLOROETHYLENE | 0 | 8120 | 5340 | 7670 | 6960 | 8650 | 7348 | 1282.33 | 17.5 | . |
| 1,1-DICHLOROETHANE | 0 | 153000 | 157000 | 164000 | 178000 | 201000 | 170600 | 19475.63 | 11.4 | . |
| TRANS-1,2-DICHLOROETHYLENE | 0 | 55500 | 51900 | 54800 | 55800 | 63500 | 56300 | 4311.03 | 7.7 | . |
| CHLOROFORM | 0 | 17200 | 15300 | 16800 | 19600 | 23400 | 18460 | 3163.54 | 17.1 | . |
| 1,2-DICHLOROETHANE | 0 | 87600 | 85200 | 105000 | 105000 | 101000 | 96760 | 9634.73 | 10.0 | . |
| 1,1,1-TRICHLOROETHANE | 0 | 19300 | 24800 | 21400 | 26600 | 26600 | 23740 | 3266.19 | 13.8 | . |
| CARBON TETRACHLORIDE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| BROMODICHLOROMETHANE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| 1,2-DICHLOROPROPANE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| TRANS-1,3-DICHLOROPROPENE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| TRICHLOROETHYLENE | 0 | 148000 | 140000 | 138000 | 152000 | 169000 | 149400 | 12361.23 | 8.3 | . |
| DIBROMOCHLOROMETHANE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| CIS-1,3-DICHLOROPROPENE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| 1,1,2-TRICHLOROETHANE | 0 | 234000 | 262000 | 266000 | 282000 | 294000 | 267600 | 22733.24 | 8.5 | . |
| BENZENE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| BROMOFORM | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| 1,1,2,2-TETRACHLOROETHENE | 0 | 183000 | 132000 | 121000 | 132000 | 154000 | 144400 | 24684.00 | 17.1 | . |
| 1,1,2,2-TETRACHLOROETHANE | 0 | 142000 | 102000 | 94400 | 102000 | 117000 | 111480 | 18930.19 | 17.0 | . |
| TOLUENE | 0 | 646 | 486 | 596 | 325 | 412 | 493 | 131.20 | 26.6 | . |
| CHLOROBENZENE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| ETHYLBENZENE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |

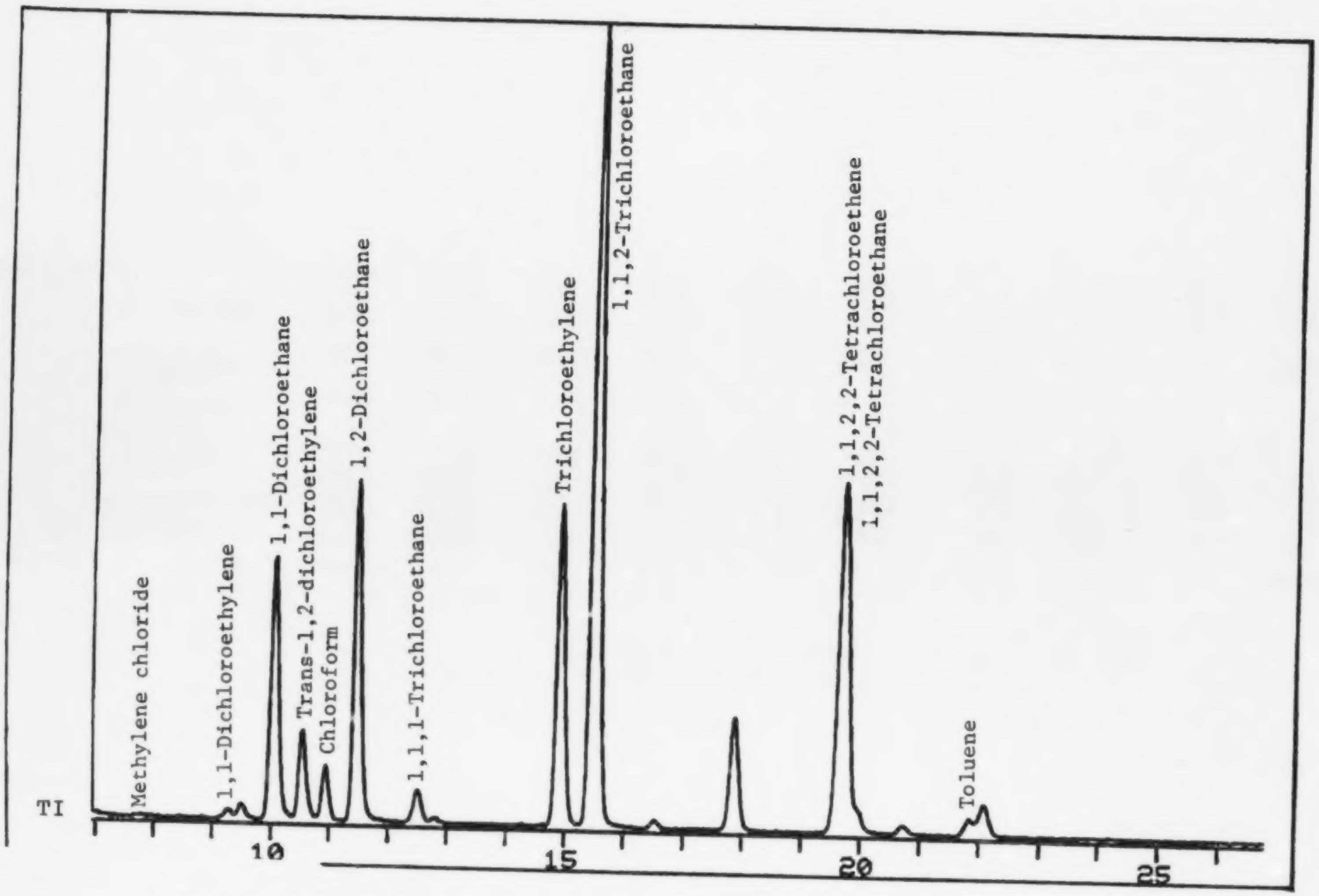

Figure II-7. GC/MS chromatogram of purgeable organics by purge and trap—Organic Still Bottoms Leachate, unspiked

## TABLE II-11. PURGEABLE ORGANICS DATA— ORGANIC STILL BOTTOMS LEACHATE, SPIKE LEVEL I

| COMPOUND | ADDED | NO_1 | NO_2 | NO_3 | NO_4 | NO_5 | MEAN | S | RSD | RECOVERY |
|---|---|---|---|---|---|---|---|---|---|---|
| METHYLENE CHLORIDE | 250000 | 178000 | 162000 | 166000 | 184000 | 187000 | 175400 | 10990.91 | 6.3 | 70 |
| 1,1-DICHLOROETHYLENE | 0 | 8960 | 8030 | 6250 | 9730 | 8030 | 8200 | 1301.81 | 15.9 | . |
| 1,1-DICHLOROETHANE | 50000 | 127000 | 104000 | 113000 | 121000 | 143000 | 121600 | 14758.05 | 12.1 | N |
| TRANS-1,2-DICHLOROETHYLENE | 250000 | 135000 | 96200 | 106000 | 128000 | 157000 | 124440 | 24092.90 | 19.4 | 27 |
| CHLOROFORM | 50000 | 36200 | 31900 | 30800 | 32300 | 41600 | 34560 | 4432.04 | 12.8 | 32 |
| 1,2-DICHLOROETHANE | 250000 | 323000 | 253000 | 283000 | 297000 | 343000 | 299800 | 34945.67 | 11.7 | 81 |
| 1,1,1-TRICHLOROETHANE | 50000 | 17300 | 11400 | 15600 | 14400 | 20900 | 15920 | 3519.52 | 22.1 | N |
| CARBON TETRACHLORIDE | 250000 | 32400 | 21200 | 16600 | 21100 | 37511 | 25762.2 | 8783.37 | 34.1 | 10 |
| BROMODICHLOROMETHANE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| 1,2-DICHLOROPROPANE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| TRANS-1,3-DICHLOROPROPENE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| TRICHLOROETHYLENE | 250000 | 151000 | 132000 | 132000 | 151000 | 167000 | 146600 | 14842.51 | 10.1 | N |
| DIBROMOCHLOROMETHANE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| CIS-1,3-DICHLOROPROPENE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| 1,1,2-TRICHLOROETHANE | 50000 | 317000 | 309000 | 323000 | 312000 | 336000 | 319400 | 10691.12 | 3.3 | 104 |
| BENZENE | 250000 | 109000 | 59800 | 66200 | 66700 | 109000 | 82140 | 24670.18 | 30.0 | 33 |
| BROMOFORM | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| 1,1,2,2-TETRACHLOROETHENE | 50000 | 20300 | 15800 | 13200 | 17300 | 16500 | 16620 | 2568.46 | 15.5 | N |
| 1,1,2,2-TETRACHLOROETHANE | 50000 | 23300 | 19000 | 18200 | 21400 | 19600 | 20300 | 2049.39 | 10.1 | N |
| TOLUENE | 250000 | 52500 | 33700 | 36100 | 32800 | 58100 | 42640 | 11787.20 | 27.6 | 17 |
| CHLOROBENZENE | 250000 | 56700 | 35800 | 38300 | 34400 | 48800 | 42800 | 9604.95 | 22.4 | 17 |
| ETHYLBENZENE | 250000 | 42200 | 24600 | 25200 | 20600 | 29900 | 28500 | 8339.06 | 29.3 | 11 |

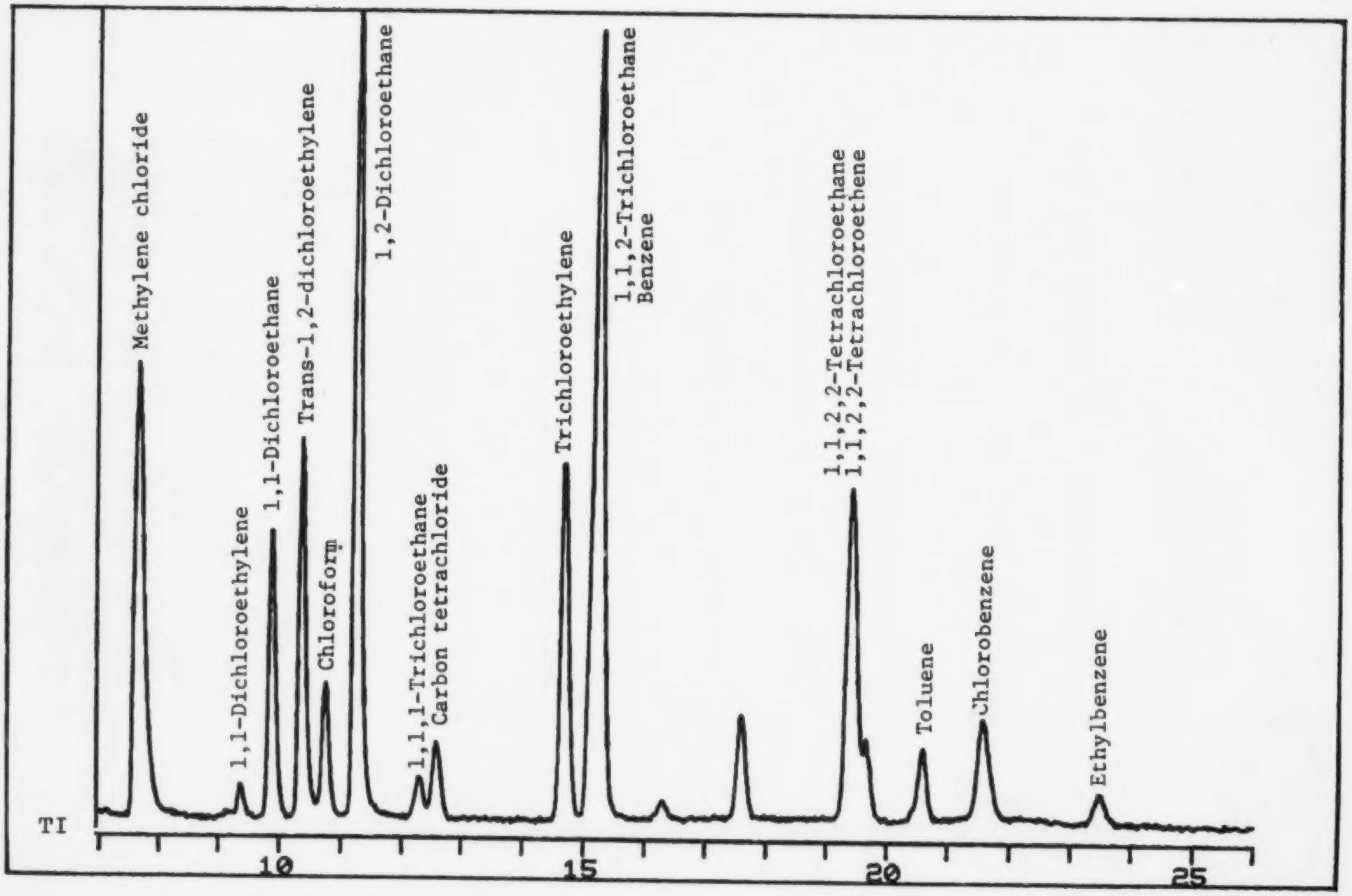

Figure II-8. GC/MS chromatogram of purgeable organics by purge and trap—Organic Still Bottoms Leachate, spiked

TABLE II-12. BASE/NEUTRAL EXTRACTABLE SEMIVOLATILE ORGANICS DATA—ORGANIC STILL BOTTOMS LEACHATE, SPIKE LEVEL 0

| COMPOUND | ADDED | NO_1 | NO_2 | NO_3 | NO_4 | NO_5 | MEAN | S | RSD | RECOVERY |
|---|---|---|---|---|---|---|---|---|---|---|
| 1,3-DICHLOROBENZENE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| 1,4-DICHLOROBENZENE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| 1,2-DICHLOROBENZENE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| HEXACHLOROETHANE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| BIS(2-CHLOROETHYL) ETHER | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| BIS(2-CHLOROISOPROPYL) ETHER | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| HEXACHLOROBUTADIENE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| NITROBENZENE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| NAPHTHALENE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| 1,2,4-TRICHLOROBENZENE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| BIS(2-CHLOROETHOXY)METHANE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| N-NITROSODI-N-PROPYLAMINE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| HEXACHLOROCYCLOPENTADIENE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| 2-CHLORONAPHTHALENE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| ISOPHORONE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| ACENAPHTHYLENE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| ACENAPHTHENE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| DIMETHYL PHTHALATE | 0 | 13 | (30) | <10 | <10 | <10 | <10.75 | 1.50 | 14.0 | . |
| 2,6-DINITROTOLUENE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| FLUORENE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| 2,4-DINITROTOLUENE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| 1,2-DIPHENYLHYDRAZINE | 0 | 46 | 26 | 76 | 26 | 31 | 41 | 21.21 | 51.7 | . |
| 4-CHLOROPHENYL PHENYL ETHER | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| DIETHYL PHTHALATE | 0 | 546 | 592 | 339 | 132 | 114 | 344.6 | 223.70 | 64.9 | . |
| N-NITROSODIPHENYLAMINE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| HEXACHLOROBENZENE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| 4-BROMOPHENYL PHENYL ETHER | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| PHENANTHRENE/ANTHRACENE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| DI-N-BUTYL PHTHALATE | 0 | <10 | <10 | <10 | <10 | <10 | <10 | Z | . | . |
| FLUORANTHENE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| PYRENE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| BENZIDINE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| BUTYLBENZYL PHTHALATE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| BIS(2-ETHYLHEXYL) PHTHALATE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| CHRYSENE/BENZO(A)ANTHRACENE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| 3,3'-DICHLOROBENZIDINE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| DI-N-OCTYL PHTHALATE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| BENZO(B)/BENZO(K)FLUORANTHENES | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| BENZO(A)PYRENE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| INDENO(1,2,3-CD)PYRENE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| DIBENZO(AH)ANTHRACENE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| BENZO(GHI)PERYLENE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |

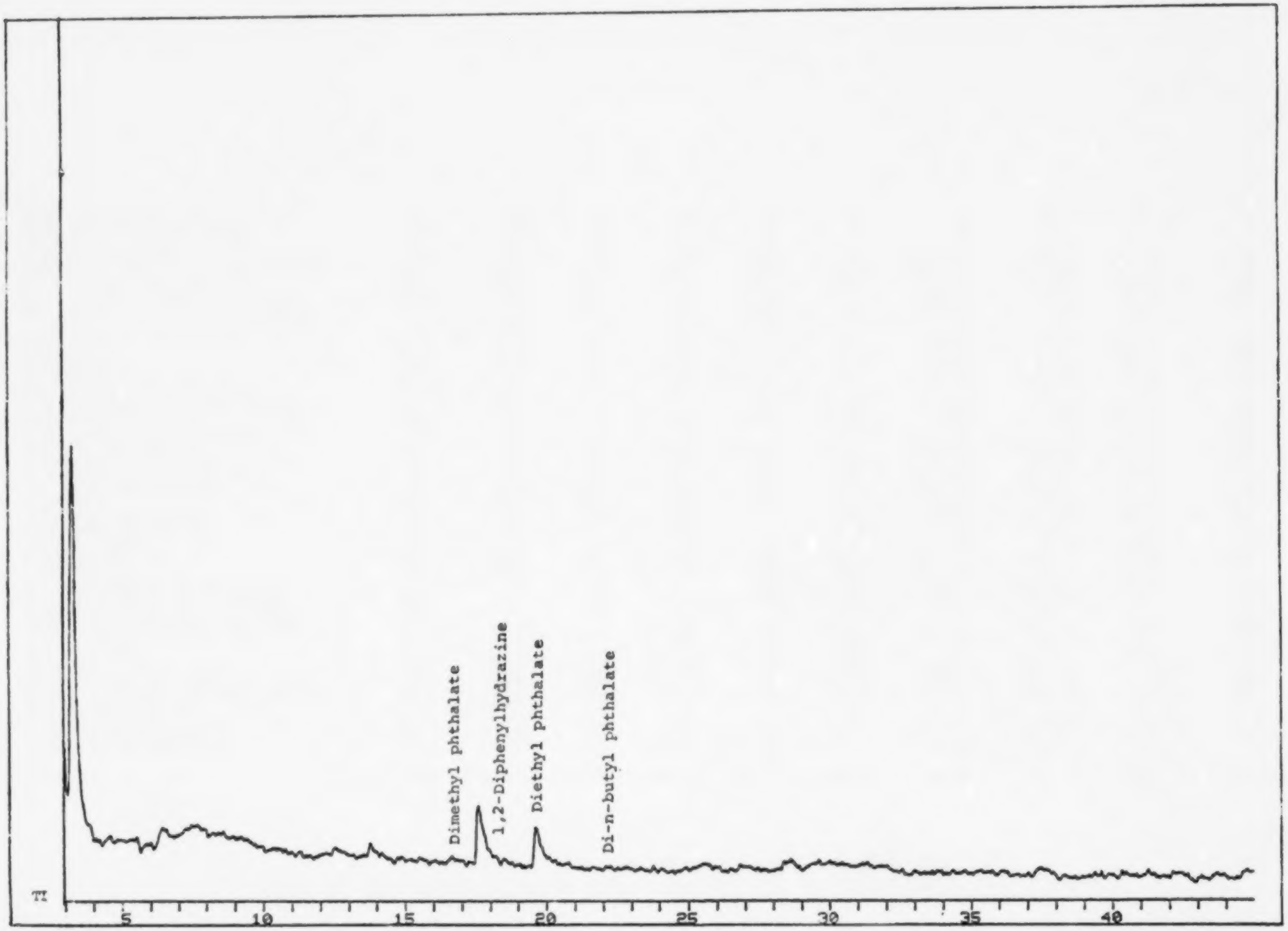

Figure II-9. GC/MS chromatogram of base/neutral extractables — Organic Still Bottoms Leachate, unspiked

## TABLE II-13. BASE/NEUTRAL EXTRACTABLE SEMIVOLATILE ORGANICS DATA— ORGANIC STILL BOTTOMS LEACHATE, SPIKE LEVEL I

| COMPOUND | ADDED | NO_1 | NO_2 | NO_3 | NO_4 | NO_5 | MEAN | S | RSD | RECOVERY |
|---|---|---|---|---|---|---|---|---|---|---|
| 1,3-DICHLOROBENZENE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| 1,4-DICHLOROBENZENE | 500 | (708) | 286 | 364 | 282 | 397 | 332.25 | 57.34 | 17.3 | 66 |
| 1,2-DICHLOROBENZENE | 100 | 80 | 74 | 83 | 84 | (123) | 80.25 | 4.50 | 5.6 | 80 |
| HEXACHLOROETHANE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| BIS(2-CHLOROETHYL) ETHER | 100 | 200 | 215 | 192 | 173 | 276 | 211.2 | 39.26 | 18.6 | 211 |
| BIS(2-CHLOROISOPROPYL) ETHER | 500 | 518 | 521 | 475 | 579 | 596 | 537.8 | 49.25 | 9.2 | 108 |
| HEXACHLOROBUTADIENE | 500 | 445 | 344 | 297 | 287 | 441 | 362.8 | 76.32 | 21.0 | 73 |
| NITROBENZENE | 100 | 143 | 170 | 117 | 136 | 169 | 147 | 22.64 | 15.4 | 147 |
| NAPHTHALENE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| 1,2,4-TRICHLOROBENZENE | 100 | 212 | 202 | 204 | 169 | 247 | 206.8 | 27.85 | 13.5 | 207 |
| BIS(2-CHLOROETHOXY)METHANE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| N-NITROSODI-N-PROPYLAMINE | 100 | <10 | <10 | <10 | <10 | <10 | <10 | Z | . | 10 |
| HEXACHLOROCYCLOPENTADIENE | 100 | 73 | 39 | 67 | 65 | 110 | 70.8 | 25.50 | 36.0 | 71 |
| 2-CHLORONAPHTHALENE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| ISOPHORONE | 500 | 534 | 450 | 461 | 480 | 631 | 511.2 | 74.35 | 14.5 | 102 |
| ACENAPHTHYLENE | 100 | 100 | 91 | 85 | 84 | (140) | 90 | 7.35 | 8.2 | 90 |
| ACENAPHTHENE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| DIMETHYL PHTHALATE | 100 | 97 | 91 | 91 | 97 | 119 | 99 | 11.58 | 11.7 | 88 |
| 2,6-DINITROTOLUENE | 100 | 49 | 51 | 84 | 77 | 108 | 73.8 | 24.59 | 33.3 | 74 |
| FLUORENE | 500 | 503 | 454 | 471 | 496 | 616 | 508 | 63.48 | 12.5 | 102 |
| 2,4-DINITROTOLUENE | 500 | 409 | 254 | 436 | 269 | 371 | 347.8 | 82.27 | 23.7 | 70 |
| 1,2-DIPHENYLHYDRAZINE | 0 | 77 | 77 | 71 | 85 | 95 | 81 | 9.27 | 11.4 | . |
| 4-CHLOROPHENYL PHENYL ETHER | 100 | 206 | 149 | 205 | 171 | 208 | 187.8 | 26.57 | 14.1 | 188 |
| DIETHYL PHTHALATE | 500 | 920 | 882 | 1340 | 1320 | 1410 | 1174.4 | 252.16 | 21.5 | 166 |
| N-NITROSODIPHENYLAMINE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| HEXACHLOROBENZENE | 500 | 593 | 530 | 742 | 636 | 784 | 657 | 104.90 | 16.0 | 131 |
| 4-BROMOPHENYL PHENYL ETHER | 500 | 600 | 535 | 713 | 514 | 722 | 616.8 | 97.29 | 15.8 | 123 |
| PHENANTHRENE/ANTHRACENE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| DI-N-BUTYL PHTHALATE | 100 | 97 | 95 | 80 | 122 | 132 | 105.2 | 21.25 | 20.2 | 95 |
| FLUORANTHENE | 500 | 523 | 466 | 567 | 583 | 654 | 558.6 | 70.32 | 12.5 | 112 |
| PYRENE | 100 | 100 | 82 | 86 | 110 | 122 | 100 | 16.61 | 16.6 | 100 |
| BENZIDINE | 100 | 18 | 18 | 17 | 15 | <10 | <15.6 | 3.36 | 21.5 | 16 |
| BUTYLBENZYL PHTHALATE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| BIS(2-ETHYLHEXYL) PHTHALATE | 0 | <10 | (211) | 13 | 14 | <10 | <11 | 2.00 | 18.2 | . |
| CHRYSENE/BENZO(A)ANTHRACENE | 500 | 1010 | 638 | 710 | 742 | 898 | 799.6 | 151.17 | 18.9 | 160 |
| 3,3'-DICHLOROBENZIDINE | 500 | 35 | 166 | 215 | 193 | 274 | 176.6 | 88.59 | 50.2 | 35 |
| DI-N-OCTYL PHTHALATE | 500 | 1050 | 635 | 714 | 811 | 802 | 802.4 | 155.90 | 19.4 | 160 |
| BENZO(B)/BENZO(K)FLUORANTHENES | 100 | 248 | 153 | 158 | 214 | (1170) | 193.25 | 45.79 | 23.7 | 193 |
| BENZO(A)PYRENE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| INDENO(1,2,3-CD)PYRENE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| DIBENZO(AH)ANTHRACENE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| BENZO(GHI)PERYLENE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |

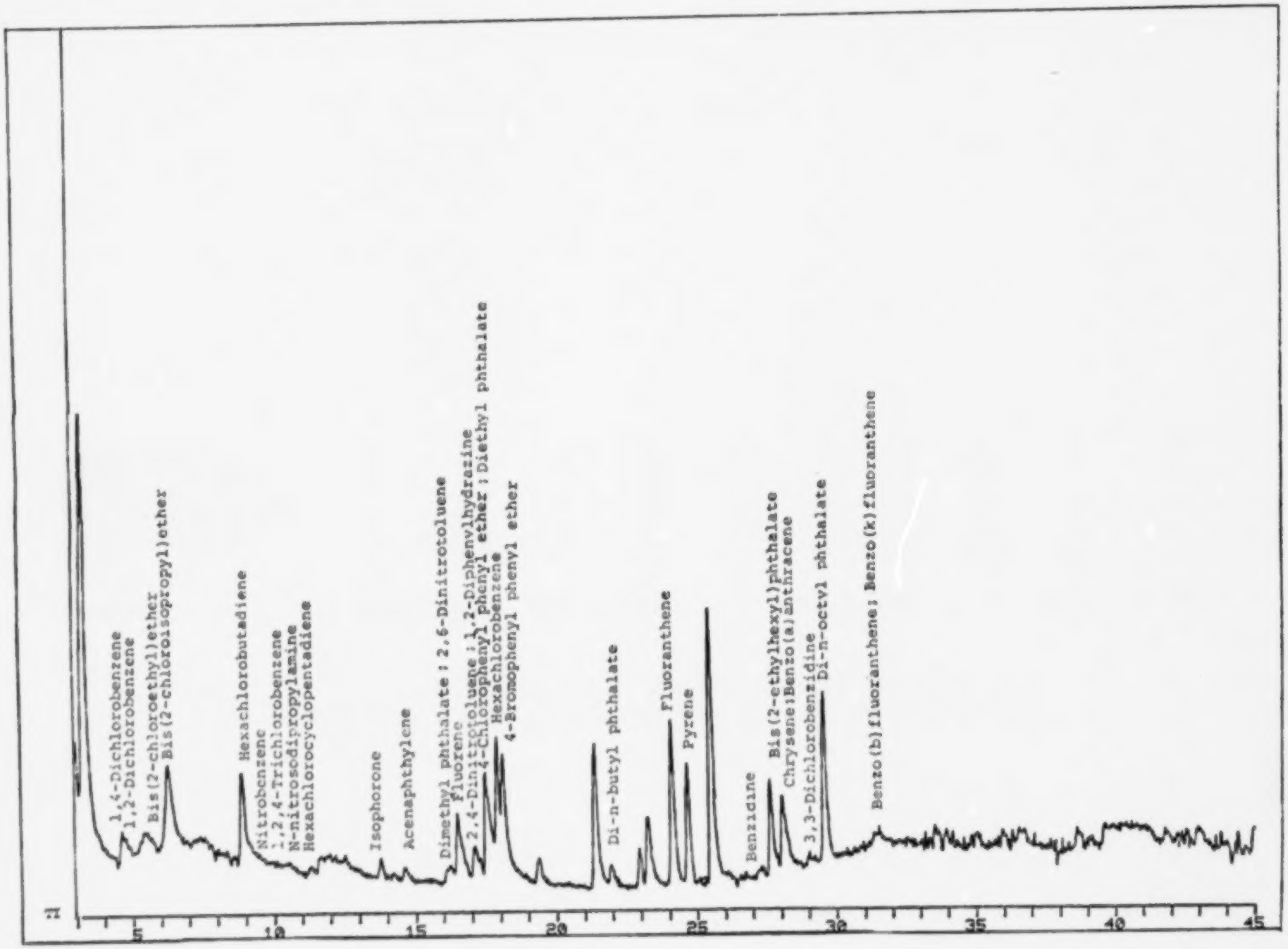

Figure II-10. GC/MS chromatogram of base/neutral extractables—Organic Still Bottoms Leachate, spiked

## TABLE II-14. ACID EXTRACTABLE SEMIVOLATILE ORGANICS DATA— ORGANIC STILL BOTTOMS LEACHATE, SPIKE LEVEL 0

| COMPOUND | ADDED | NO_1 | NO_2 | NO_3 | NO_4 | NO_5 | MEAN | S | RSD | RECOVERY |
|---|---|---|---|---|---|---|---|---|---|---|
| 2-CHLOROPHENOL | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| 2-NITROPHENOL | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| PHENOL | 0 | 231 | 236 | 217 | 174 | 117 | 195 | 49.96 | 25.6 | . |
| 2,4-DIMETHYLPHENOL | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| 2,4-DICHLOROPHENOL | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| 2,4,6-TRICHLOROPHENOL | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| 4-CHLORO-3-METHYLPHENOL | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| 2,4-DINITROPHENOL | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| 4,6-DINITRO-O-CRESOL | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| PENTACHLOROPHENOL | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| 4-NITROPHENOL | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |

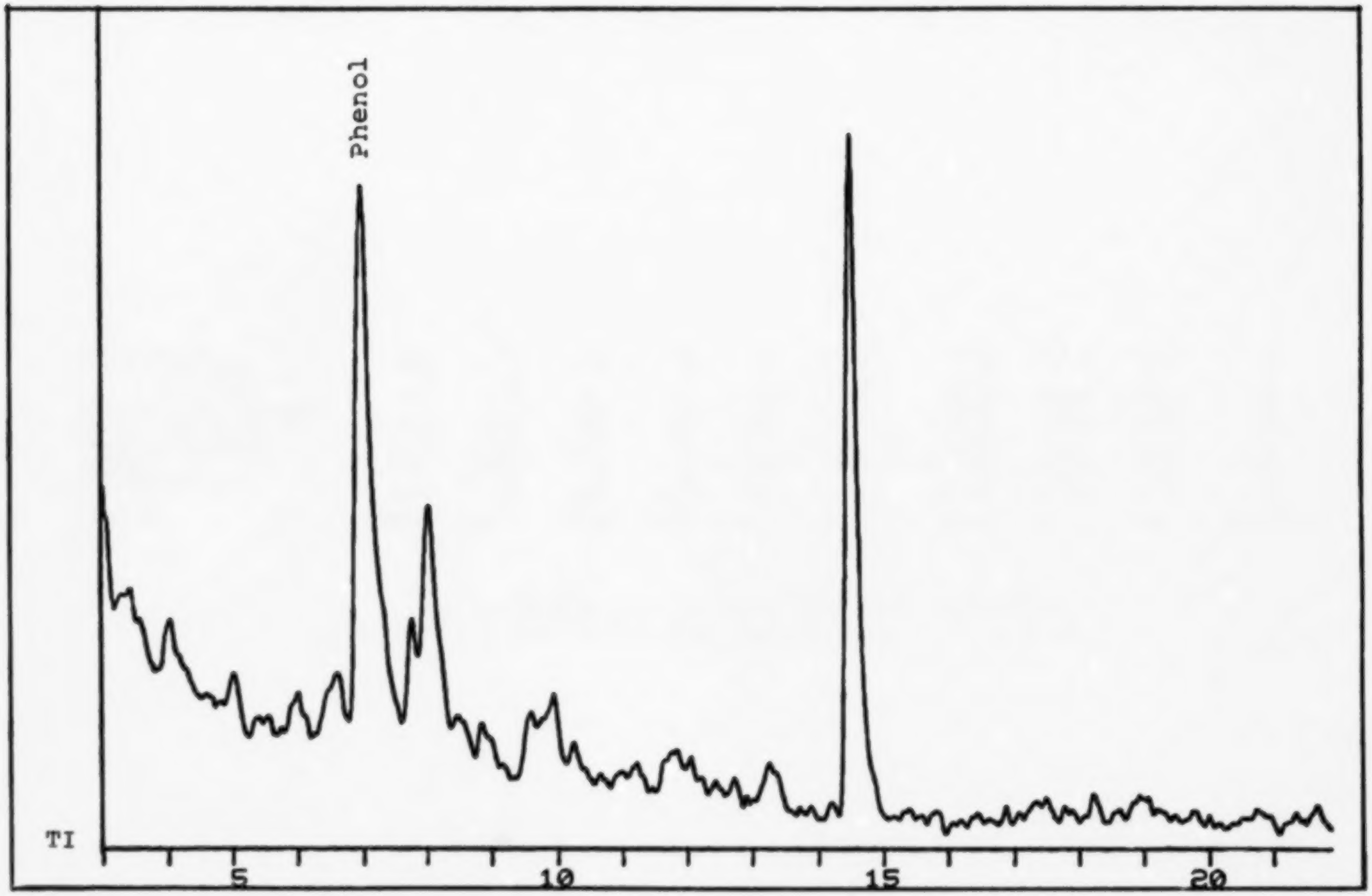

Figure II-11. GC/MS chromatogram of acid extractables—
Organic Still Bottoms Leachate, unspiked

## TABLE II-15. ACID EXTRACTABLE SEMIVOLATILE ORGANICS DATA— ORGANIC STILL BOTTOMS LEACHATE, SPIKE LEVEL I

| COMPOUND | ADDED | NO_1 | NO_2 | NO_3 | NO_4 | NO_5 | MEAN | S | RSD | RECOVERY |
|---|---|---|---|---|---|---|---|---|---|---|
| 2-CHLOROPHENOL | 500 | 178 | 187 | 145 | 166 | 231 | 181.4 | 31.88 | 17.6 | 36 |
| 2-NITROPHENOL | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| PHENOL | 1000 | 1450 | 1450 | 1410 | (1100) | 1483 | 1448.25 | 29.87 | 2.1 | 125 |
| 2,4-DIMETHYLPHENOL | 100 | 40 | 47 | 27 | <25 | <25 | <32.8 | 10.11 | 30.8 | 33 |
| 2,4-DICHLOROPHENOL | 100 | <25 | <25 | <25 | <25 | <25 | <25 | Z | . | 25 |
| 2,4,6-TRICHLOROPHENOL | 500 | 108 | 113 | 110 | 81 | 87 | 99.8 | 14.69 | 14.7 | 20 |
| 4-CHLORO-3-METHYLPHENOL | 0 | 77 | 102 | 70 | 70 | 99 | 83.6 | 15.73 | 18.8 | . |
| 2,4-DINITROPHENOL | 500 | 243 | 202 | 169 | 193 | 233 | 207.4 | 30.53 | 14.7 | 41 |
| 4,6-DINITRO-O-CRESOL | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| PENTACHLOROPHENOL | 100 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | 0 |
| 4-NITROPHENOL | 100 | 47 | 37 | 60 | 34 | 53 | 46.2 | 10.85 | 23.5 | 46 |

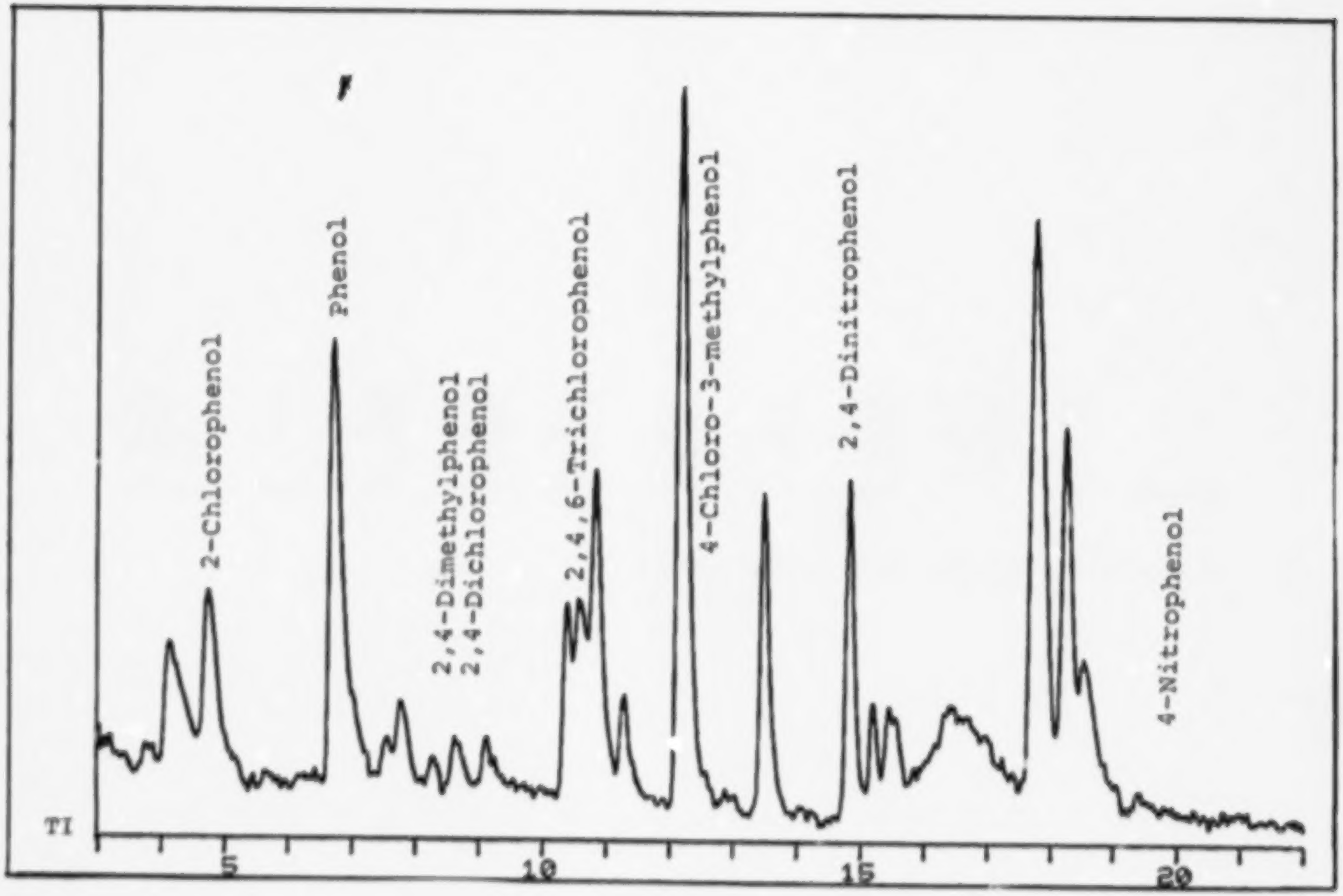

Figure II-12. GC/MS chromatogram of acid extractables — Organic Still Bottoms Leachate, spiked

TABLE II-16. METALS DATA—
ORGANIC STILL BOTTOMS LEACHATE, SPIKE LEVEL 0

| ELEMENT | ADDED | NO_1 | NO_2 | NO_3 | NO_4 | NO_5 | MEAN | S | RSD | RECOVERY |
|---|---|---|---|---|---|---|---|---|---|---|
| CADMIUM | 0 | 12 | 16 | 12 | 12 | 12 | 12.8 | 1.79 | 14.0 | . |
| CHROMIUM | 0 | 21 | 21 | 21 | (29) | 21 | 21 | 0.00 | 0.0 | . |
| COPPER | 0 | 23600 | 23200 | 23800 | 23600 | 24000 | 23640 | 296.65 | 1.3 | . |
| LEAD | 0 | 268 | 268 | 293 | 268 | 243 | 268 | 17.68 | 6.6 | . |
| NICKEL | 0 | 1480 | 1460 | 1440 | 1460 | 1400 | 1448 | 30.33 | 2.1 | . |
| ZINC | 0 | 6090 | 5650 | 5560 | 5560 | 6090 | 5790 | 276.32 | 4.8 | . |

## TABLE II-17. METALS DATA—
## ORGANIC STILL BOTTOMS LEACHATE, SPIKE LEVEL I

| ELEMENT | ADDED | NO_1 | NO_2 | NO_3 | NO_4 | NO_5 | MEAN | S | RSD | RECOVERY |
|---|---|---|---|---|---|---|---|---|---|---|
| CADMIUM | 200 | 227 | 231 | 223 | 219 | 219 | 223.8 | 5.22 | 2.3 | 105 |
| CHROMIUM | 200 | 118 | 118 | 125 | 125 | 125 | 122.2 | 3.83 | 3.1 | 51 |
| COPPER | 25000 | 47900 | 47400 | 47900 | 48700 | 47900 | 47960 | 466.90 | 1.0 | 97 |
| LEAD | 1000 | 1380 | 1360 | 1380 | 1360 | 1360 | 1368 | 10.95 | 0.8 | 110 |
| NICKEL | 2000 | 3160 | 3210 | 3250 | 3230 | 3250 | 3220 | 37.42 | 1.2 | 89 |
| ZINC | 4000 | 10900 | 10300 | 10200 | 10300 | 10600 | 10460 | 288.10 | 2.8 | 117 |

### TABLE II-18. METALS DATA— ORGANIC STILL BOTTOMS LEACHATE, SPIKE LEVEL II

| ELEMENT | ADDED | NO_1 | NO_2 | NO_3 | NO_4 | NO_5 | MEAN | S | RSD | RECOVERY |
|---|---|---|---|---|---|---|---|---|---|---|
| CADMIUM | 1000 | 1010 | 1020 | 995 | 1070 | 1080 | 1035 | 37.75 | 3.6 | 102 |
| CHROMIUM | 1000 | 479 | 472 | 487 | 479 | 479 | 479.2 | 5.31 | 1.1 | 46 |
| COPPER | 25000 | 49000 | 49000 | 49000 | 49000 | 49000 | 49000 | 0.00 | 0.0 | 101 |
| LEAD | 5000 | 5570 | 5570 | 5600 | 5570 | 5500 | 5562 | 37.01 | 0.7 | 106 |
| NICKEL | 15000 | 18400 | 18000 | 17700 | 17700 | 17500 | 17860 | 350.71 | 2.0 | 109 |
| ZINC | 35000 | 45200 | 44700 | 44700 | 44600 | 45200 | 44880 | 294.96 | 0.7 | 112 |

# Appendix III. Total Content Interlaboratory Study Data

## Legend for Verification Analyses and Quality Assurance for Total Content Interlaboratory Study Data*

| Symbols | Identification |
|---|---|
| . | Not determined. |
| < | Lower detection limit of method. |
| (   ) | Found to be a statistical outlier and not included in the calculations. |
| Z | Standard deviation was not calculated because all points in the data set were less than lower detection limit of method and/or zero. |
| N | Percent recovery was calculated to be less than zero. |

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* Concentrations are reported as microgram of compound per gram of residual waste (parts per million).

## A. Ink Pigment Waste (Interlaboratory)

## TABLE III-1. PURGEABLE ORGANICS DATA— INK PIGMENT WASTE (INTERLABORATORY), SPIKE LEVEL 0

| COMPOUND | ADDED | NO_1 | NO_2 | NO_3 | NO_4 | NO_5 | MEAN | S | RSD | RECOVERY |
|---|---|---|---|---|---|---|---|---|---|---|
| METHYLENE CHLORIDE | 0 | 8.2 | 7.4 | 5.8 | 4.6 | 3.8 | 5.96 | 1.85 | 31.0 | . |
| 1,1-DICHLOROFTHYLENE | 0 | 1.1 | 0.8 | 0.45 | 0.66 | 0.45 | 0.692 | 0.27 | 39.3 | . |
| 1,1-DICHLOROETHANE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| TRANS-1,2-DICHLOROETHYLENE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| CHLOROFORM | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| 1,2-DICHLOROETHANE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| 1,1,1-TRICHLOROETHANE | 0 | 0.39 | 0.4 | 0.34 | 0.35 | 0.25 | 0.346 | 0.06 | 17.2 | . |
| CARBON TETRACHLORIDE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| BROMODICHLOROMETHANE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| 1,2-DICHLOROPROPANE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| TRANS-1,3-DICHLOROPROPENE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| TRICHLOROETHYLENE | 0 | 9.4 | 8.1 | 5.5 | 5 | 4.3 | 6.46 | 2.18 | 33.8 | . |
| DIBROMOCHLOROMETHANE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| CIS-1,3-DICHLOROPROPENE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| 1,1,2-TRICHLOROETHANE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| BENZENE | 0 | 3.9 | 3.3 | 2.8 | 2.5 | 1.7 | 2.84 | 0.83 | 29.2 | . |
| BROMOFORM | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| 1,1,2,2-TETRACHLOROETHENE | 0 | 5.8 | 4.5 | 3.7 | 4 | 2.4 | 4.08 | 1.24 | 30.3 | . |
| 1,1,2,2-TETRACHLOROETHANE | 0 | 4.3 | 3.6 | 2.9 | 2.7 | 1.8 | 3.06 | 0.94 | 30.9 | . |
| TOLUENE | 0 | 63 | 64 | 51 | 48 | 36 | 52.4 | 11.59 | 22.1 | . |
| CHLOROBENZENE | 0 | 2.7 | 3 | 2.7 | 1.8 | 1.4 | 2.32 | 0.68 | 29.5 | . |
| ETHYLBENZENE | 0 | 71 | 80 | 56 | 46 | 33 | 57.2 | 18.86 | 33.0 | . |

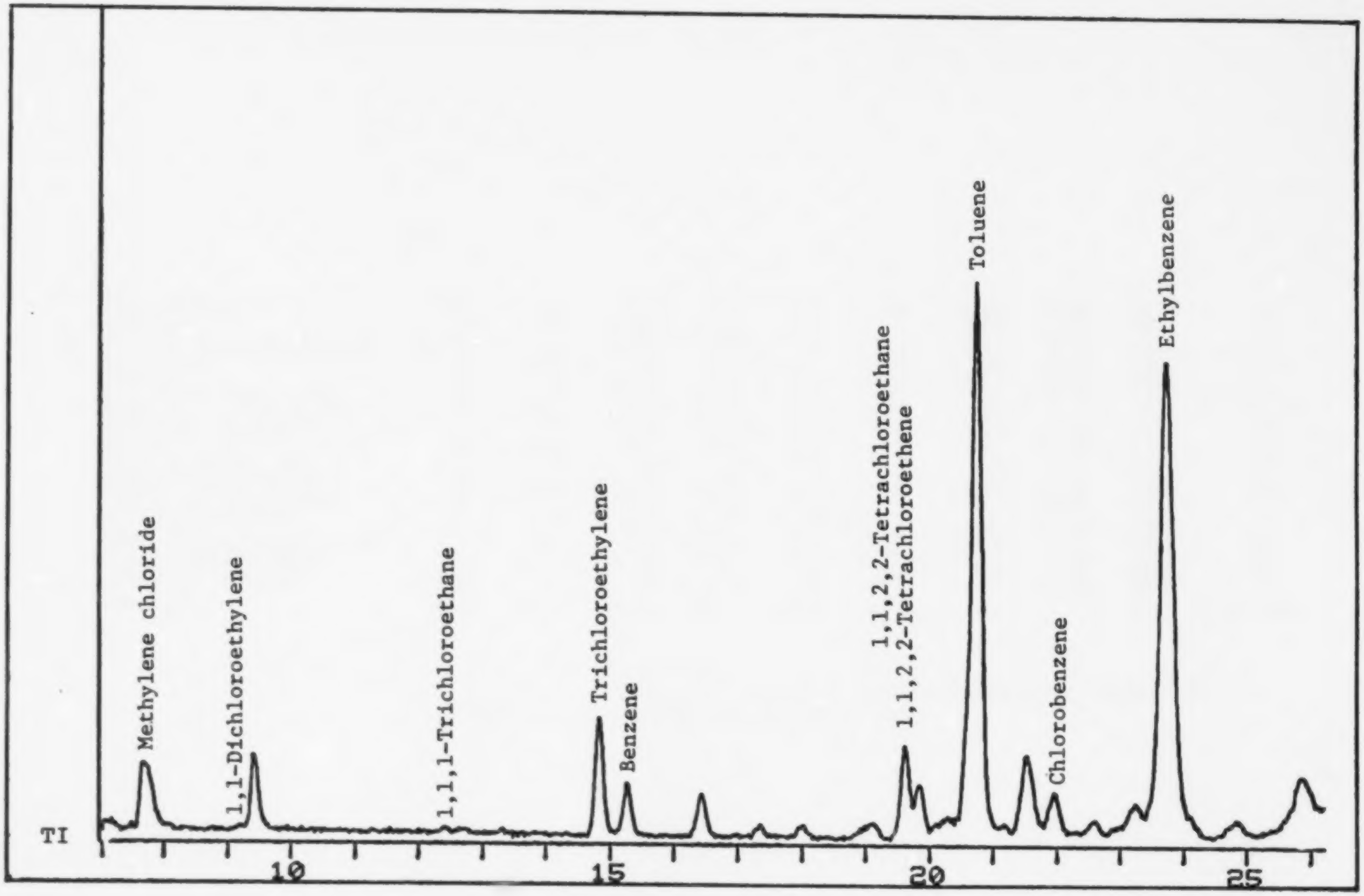

Figure III-1. GC/MS chromatogram of purgeable organics by purge and trap—Ink Pigment Waste (Interlaboratory), unspiked

TABLE III-2. PURGEABLE ORGANICS DATA—
INK PIGMENT WASTE (INTERLABORATORY), SPIKE LEVEL I

| COMPOUND | ADDED | NO_1 | NO_2 | NO_3 | NO_4 | NO_5 | MEAN | S | RSD | RECOVERY |
|---|---|---|---|---|---|---|---|---|---|---|
| METHYLENE CHLORIDE | 1.9 | 5 | (12) | 4.1 | 3 | 4.5 | 4.15 | 0.85 | 20.5 | N |
| 1,1-DICHLOROETHYLENE | 1.9 | 1.2 | 1.4 | 1 | 1.7 | 0.87 | 1.234 | 0.33 | 26.7 | 29 |
| 1,1-DICHLOROETHANE | 2 | 0.92 | 0.72 | 1.2 | 1.1 | 0.84 | 0.956 | 0.19 | 20.3 | 48 |
| TRANS-1,2-DICHLOROETHYLENE | 2 | 0.97 | 0.66 | 0.72 | 1.3 | 0.81 | 0.892 | 0.26 | 28.7 | 45 |
| CHLOROFORM | 1.9 | 1 | 0.64 | 1.1 | 1.5 | 1.2 | 1.088 | 0.31 | 28.7 | 57 |
| 1,2-DICHLOROETHANE | 2 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | 0 |
| 1,1,1-TRICHLOROETHANE | 2 | 1.3 | 0.87 | 1.3 | 1.5 | 1.1 | 1.214 | 0.24 | 19.7 | 43 |
| CARBON TETRACHLORIDE | 2 | 0.94 | 0.41 | 0.92 | 1.3 | 0.84 | 0.882 | 0.32 | 36.0 | 44 |
| BROMODICHLOROMETHANE | 1.9 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | 0 |
| 1,2-DICHLOROPROPANE | 2 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | 0 |
| TRANS-1,3-DICHLOROPROPENE | 2 | 0.95 | 1.1 | 1.1 | 1.6 | 1.1 | 1.17 | 0.25 | 21.3 | 58 |
| TRICHLOROETHYLENE | 1.9 | 5.3 | (12) | 5.9 | 5.7 | 6.3 | 5.8 | 0.42 | 7.2 | N |
| DIBROMOCHLOROMETHANE | 2 | 0.89 | 0.91 | 0.83 | (2.9) | 0.91 | 0.885 | 0.04 | 4.3 | 44 |
| CIS-1,3-DICHLOROPROPENE | 1.6 | 0.9 | 1.2 | 1 | 1.5 | 0.85 | 1.09 | 0.27 | 24.4 | 68 |
| 1,1,2-TRICHLOROETHANE | 2 | 7.6 | (14) | 8.8 | 8.1 | 8.3 | 8.2 | 0.50 | 6.1 | 410 |
| BENZENE | 1.9 | 2.9 | (6.4) | 3.4 | 2.9 | 3.5 | 3.175 | 0.32 | 10.1 | 18 |
| BROMOFORM | 2 | 0.93 | 1.3 | 1.2 | 1.6 | 1.1 | 1.226 | 0.25 | 20.4 | 61 |
| 1,1,2,2-TETRACHLOROETHENE | 2 | 4.6 | (6.8) | 4.3 | 4 | 4.8 | 4.425 | 0.35 | 7.9 | 17 |
| 1,1,2,2-TETRACHLOROETHANE | 1.9 | 3.4 | 5.4 | 4.1 | 3 | 3.4 | 3.86 | 0.95 | 24.5 | 42 |
| TOLUENE | 2 | 41 | (88) | 55 | 47 | 56 | 49.75 | 7.09 | 14.2 | N |
| CHLOROBENZENE | 1.9 | 3.3 | 4.8 | 3.2 | 3.9 | 3.2 | 3.68 | 0.69 | 18.8 | 72 |
| ETHYLBENZENE | 1.9 | 47 | (102) | 65 | 53 | 50 | 53.75 | 7.89 | 14.7 | N |

## TABLE III-3. PURGEABLE ORGANICS DATA— INK PIGMENT WASTE (INTERLABORATORY), SPIKE LEVEL II

| COMPOUND | ADDED | NO_1 | NO_2 | NO_3 | NO_4 | NO_5 | MEAN | S | RSD | RECOVERY |
|---|---|---|---|---|---|---|---|---|---|---|
| METHYLENE CHLORIDE | 9.9 | 7.7 | 5.2 | 6.5 | 4.8 | 5 | 5.84 | 1.23 | 21.1 | N |
| 1,1-DICHLOROETHYLENE | 9.9 | 4.3 | 3.2 | 3.6 | 3.5 | 3.7 | 3.66 | 0.40 | 11.0 | 30 |
| 1,1-DICHLOROETHANE | 10 | 5.5 | 4.6 | 4.2 | 4.8 | 5 | 4.82 | 0.48 | 10.0 | 48 |
| TRANS-1,2-DICHLOROETHYLENE | 10 | 3.6 | 2.9 | 3.1 | 3.4 | 2.9 | 3.18 | 0.31 | 9.8 | 32 |
| CHLOROFORM | 10 | 6.1 | 5.2 | 4.9 | 4.9 | 5.3 | 5.28 | 0.49 | 9.3 | 53 |
| 1,2-DICHLOROETHANE | 10 | 3.1 | 2.9 | 3.1 | 3 | 3 | 3.02 | 0.08 | 2.8 | 30 |
| 1,1,1-TRICHLOROETHANE | 10 | 6.5 | 5.1 | 5.2 | 5.4 | 5.5 | 5.54 | 0.56 | 10.1 | 57 |
| CARBON TETRACHLORIDE | 10 | 5.5 | 4.5 | 4.5 | 4.5 | 4.7 | 4.74 | 0.43 | 9.1 | 47 |
| BROMODICHLOROMETHANE | 9.9 | 4.8 | 3.7 | 3.5 | 3.5 | 3.2 | 3.74 | 0.62 | 16.5 | 38 |
| 1,2-DICHLOROPROPANE | 10 | (5) | 2.9 | 2.7 | 2.6 | 3.2 | 2.85 | 0.26 | 9.3 | 28 |
| TRANS-1,3-DICHLOROPROPENE | 10 | 5.5 | 4.8 | 4.3 | 4.6 | 4.9 | 4.82 | 0.44 | 9.2 | 48 |
| TRICHLOROETHYLENE | 9.9 | 13 | 8.2 | 10 | 6.7 | 6.3 | 8.84 | 2.74 | 31.0 | 24 |
| DIBROMOCHLOROMETHANE | 10 | (5.2) | 3.9 | 3.8 | 3.9 | 3.8 | 3.85 | 0.06 | 1.5 | 38 |
| CIS-1,3-DICHLOROPROPENE | 8.1 | (4.6) | 3.5 | 3.5 | 3.5 | 3.7 | 3.55 | 0.10 | 2.8 | 44 |
| 1,1,2-TRICHLOROETHANE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| BENZENE | 10 | 7.4 | 4.9 | 5.7 | 4.5 | 4.1 | 5.32 | 1.30 | 24.5 | 25 |
| BROMOFORM | 10 | 6.2 | 1.1 | 4.9 | 5.1 | 4.1 | 4.28 | 1.93 | 45.1 | 43 |
| 1,1,2,2-TETRACHLOROETHENE | 10 | 7.8 | 4.4 | 6.6 | 3.4 | 2.5 | 4.94 | 2.21 | 44.8 | 9 |
| 1,1,2,2-TETRACHLOROETHANE | 10 | 6.2 | 3.6 | 6 | 2.7 | 2.3 | 4.16 | 1.83 | 44.1 | 11 |
| TOLUENE | 10 | 60 | 36 | 43 | 27 | 22 | 37.6 | 14.91 | 39.7 | N |
| CHLOROBENZENE | 10 | 11 | 6.9 | 7.8 | 5.3 | 3.5 | 6.9 | 2.82 | 40.8 | 46 |
| ETHYLBENZENE | 10 | 74 | 39 | 50 | 23 | 17 | 40.6 | 22.77 | 56.1 | N |

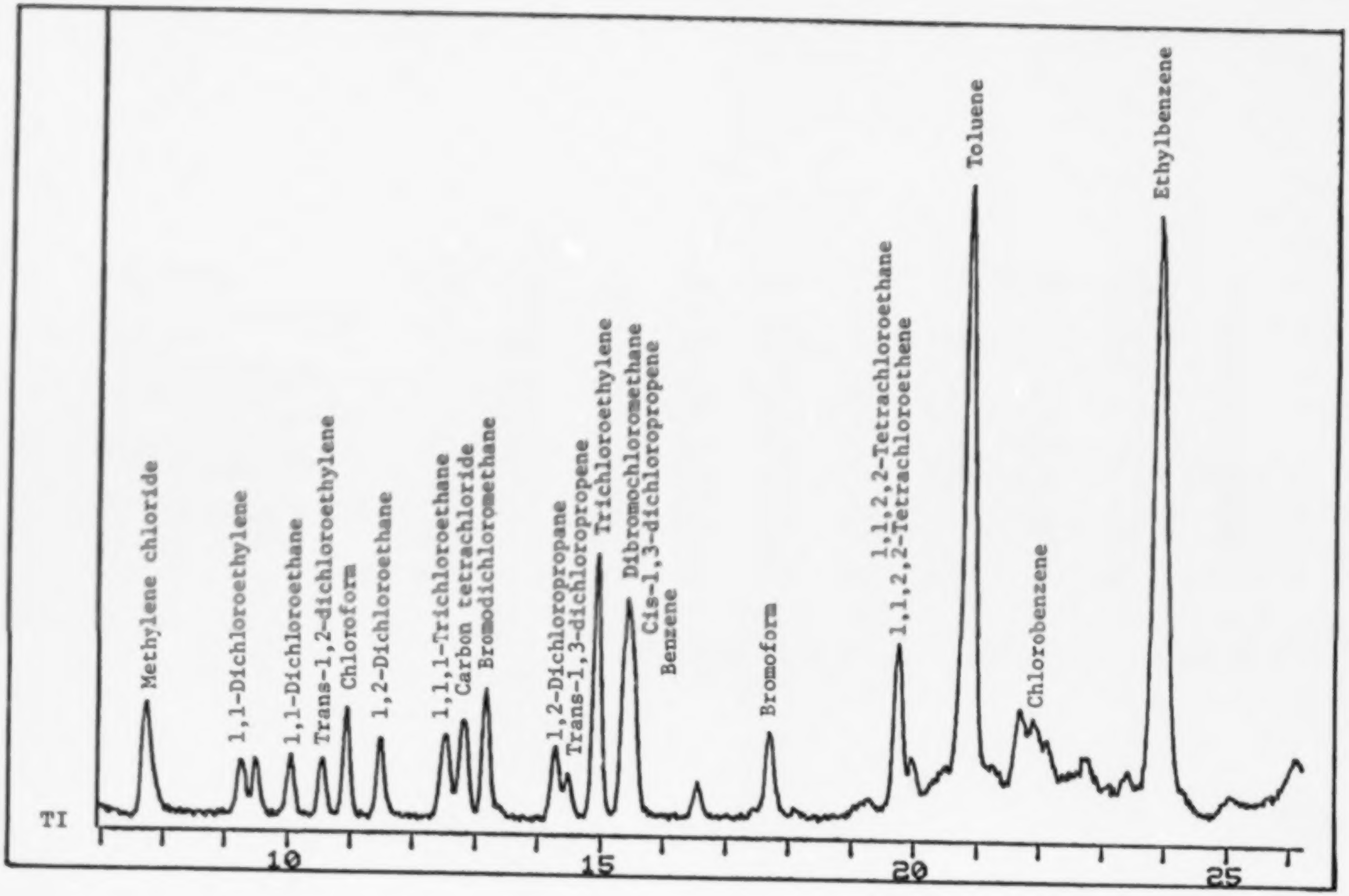

Figure III-2. GC/MS chromatogram of purgeable organics by purge and trap—Ink Pigment Waste (Interlaboratory), spiked

## TABLE III-4. BASE/NEUTRAL EXTRACTABLE SEMIVOLATILE ORGANICS DATA— INK PIGMENT WASTE (INTERLABORATORY), SPIKE LEVEL 0

| COMPOUND | ADDED | NO_1 | NO_2 | NO_3 | NO_4 | NO_5 | MEAN | S | RSD | RECOVERY |
|---|---|---|---|---|---|---|---|---|---|---|
| 1,3-DICHLOROBENZENE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| 1,4-DICHLOROBENZENE | 0 | 20 | 27 | 52 | 26 | 45 | 34 | 13.73 | 40.4 | . |
| 1,2-DICHLOROBENZENE | 0 | 24 | 33 | 65 | 29 | 50 | 40.2 | 16.96 | 42.2 | . |
| HEXACHLOROETHANE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| BIS(2-CHLOROETHYL) ETHER | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| BIS(2-CHLOROISOPROPYL) ETHER | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| HEXACHLOROBUTADIENE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| NITROBENZENE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| NAPHTHALENE | 0 | 47 | 67 | 165 | 82 | 114 | 95 | 46.15 | 48.6 | . |
| 1,2,4-TRICHLOROBENZENE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| BIS(2-CHLOROETHOXY)METHANE | 0 | 12 | 8.6 | 22 | 16 | 15 | 14.72 | 4.99 | 33.9 | . |
| N-NITROSODI-N-PROPYLAMINE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| HEXACHLOROCYCLOPENTADIENE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| 2-CHLORONAPHTHALENE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| ISOPHORONE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| ACENAPHTHYLENE | 0 | 4.7 | 11 | 36 | 13 | 23 | 17.54 | 12.24 | 69.8 | . |
| ACENAPHTHENE | 0 | 5.8 | 7.7 | 18 | 12 | 13 | 11.3 | 4.78 | 42.3 | . |
| DIMETHYL PHTHALATE | 0 | 1.5 | 1.8 | 3.5 | 2.3 | 2.9 | 2.46 | 0.92 | 37.3 | . |
| 2,6-DINITROTOLUENE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| FLUORENE | 0 | 11 | 18 | 33 | 20 | 28 | 22 | 8.63 | 39.2 | . |
| 2,4-DINITROTOLUENE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| 1,2-DIPHENYLHYDRAZINE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| 4-CHLOROPHENYL PHENYL ETHER | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| DIETHYL PHTHALATE | 0 | 1.5 | 4.2 | 4.9 | 2.5 | 4.2 | 3.46 | 1.41 | 40.7 | . |
| N-NITROSODIPHENYLAMINE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| HEXACHLOROBENZENE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| 4-BROMOPHENYL PHENYL ETHER | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| PHENANTHRENE/ANTHRACENE | 0 | 25 | 42 | 82 | 60 | 80 | 57.8 | 24.54 | 42.5 | . |
| DI-N-BUTYL PHTHALATE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| FLUORANTHENE | 0 | 7.1 | 9.3 | 18 | 11 | 13 | 11.68 | 4.15 | 35.5 | . |
| PYRENE | 0 | 4.8 | 5.7 | 12 | 6.3 | 8.4 | 7.42 | 2.88 | 38.9 | . |
| BENZIDINE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| BUTYLBENZYL PHTHALATE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| BIS(2-ETHYLHEXYL) PHTHALATE | 0 | 11 | 12 | (20) | 12 | 14 | 12.25 | 1.26 | 10.3 | . |
| CHRYSENE/BENZO(A)ANTHRACENE | 0 | 6.5 | 7 | (13) | 6.6 | 7.9 | 7 | 0.64 | 9.1 | . |
| 3,3'-DICHLOROBENZIDINE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| DI-N-OCTYL PHTHALATE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| BENZO(B)/BENZO(K)FLUORANTHENES | 0 | 5.3 | 3.2 | 8.3 | 3.4 | 3.7 | 4.78 | 2.13 | 44.7 | . |
| BENZO(A)PYRENE | 0 | 6.4 | 5.2 | (12) | 5.3 | 6.5 | 5.85 | 0.70 | 11.9 | . |
| INDENO(1,2,3-CD)PYRENE | 0 | 2.1 | 1.9 | 8.7 | <1 | 4.7 | <3.68 | 3.13 | 84.9 | . |
| DIBENZO(AH)ANTHRACENE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| BENZO(GHI)PERYLENE | 0 | 0 | (2.2) | 0 | 0 | 0 | 0 | Z | . | . |

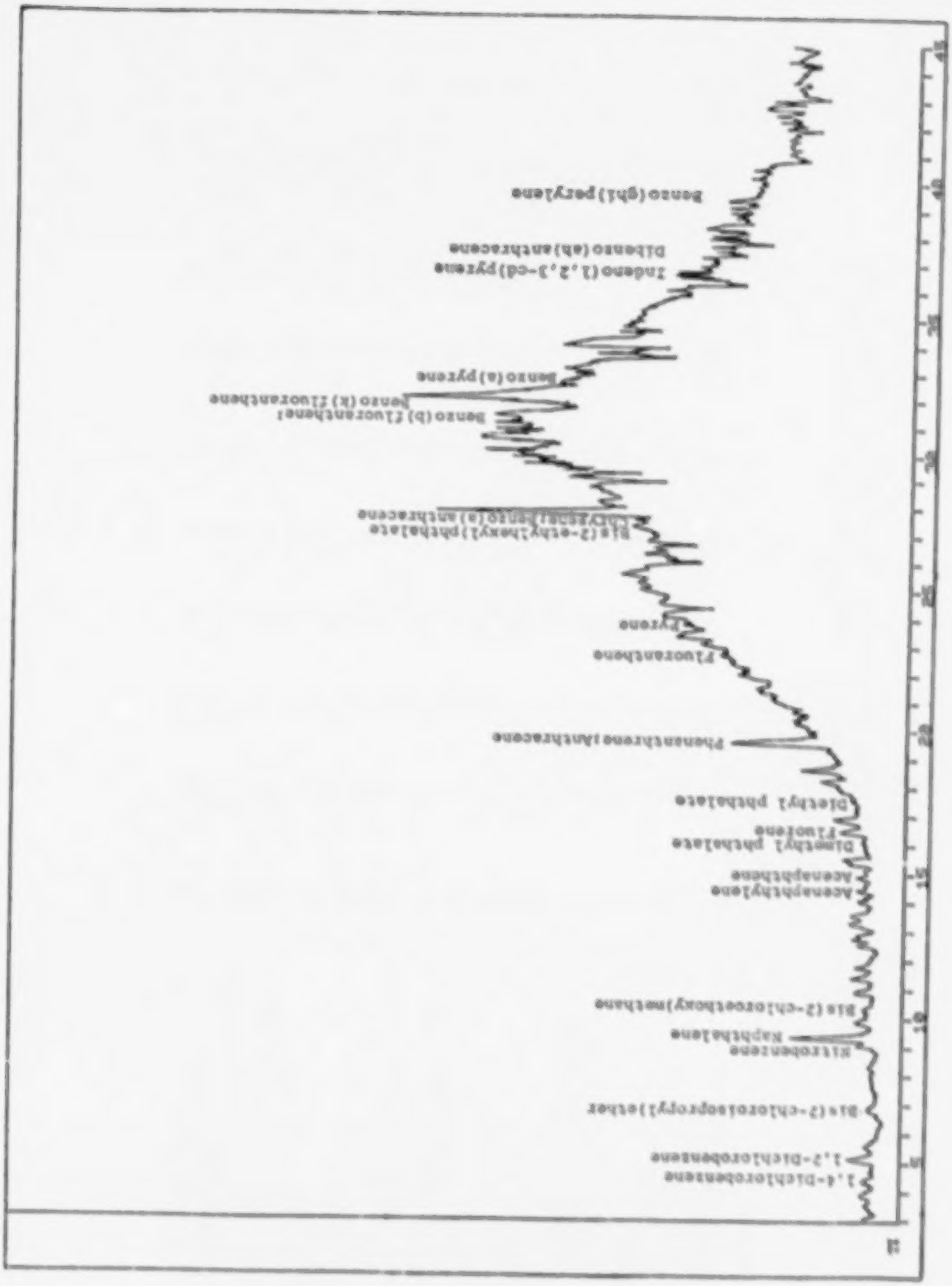

Figure III-3. GC/MS chromatogram of base/neutral extractables—Ink Pigment Waste (Interlaboratory), unspiked

TABLE III-5. BASE/NEUTRAL EXTRACTABLE SEMIVOLATILE ORGANICS DATA—
INK PIGMENT WASTE (INTERLABORATORY), SPIKE LEVEL I

| COMPOUND | ADDED | NO_1 | NO_2 | NO_3 | NO_4 | NO_5 | MEAN | S | RSD | RECOVERY |
|---|---|---|---|---|---|---|---|---|---|---|
| 1,3-DICHLOROBENZENE | 1.71 | 1.1 | 0.92 | 0.97 | . | . | 0.996667 | 0.09 | 9.3 | 58 |
| 1,4-DICHLOROBENZENE | [illegible] | [illegible] | [illegible] | 15 | . | . | 17.3333 | [illegible] | [illegible] | [illegible] |
| 1,2-DICHLOROBENZENE | [illegible] | 18 | [illegible] | [illegible] | . | . | 19.3333 | [illegible] | [illegible] | [illegible] |
| HEXACHLOROETHANE | [illegible] | 3.1 | [illegible] | [illegible] | . | . | [illegible] | [illegible] | [illegible] | 143 |
| BIS(2-CHLOROETHYL) ETHER | [illegible] | [illegible] | [illegible] | [illegible] | . | . | 1.76667 | [illegible] | [illegible] | [illegible] |
| BIS(2-CHLOROISOPROPYL) ETHER | [illegible] | [illegible] | [illegible] | [illegible] | . | . | [illegible] | [illegible] | [illegible] | [illegible] |
| HEXACHLOROBUTADIENE | [illegible] | [illegible] | [illegible] | [illegible] | . | . | [illegible] | [illegible] | [illegible] | [illegible] |
| NITROBENZENE | [illegible] | [illegible] | [illegible] | [illegible] | . | . | [illegible] | [illegible] | [illegible] | [illegible] |
| NAPHTHALENE | [illegible] | 37 | [illegible] | 31 | . | . | 37.3333 | [illegible] | [illegible] | [illegible] |
| 1,2,4-TRICHLOROBENZENE | [illegible] | [illegible] | [illegible] | [illegible] | . | . | [illegible] | [illegible] | [illegible] | [illegible] |
| BIS(2-CHLOROETHOXY)METHANE | [illegible] | [illegible] | [illegible] | [illegible] | . | . | [illegible] | [illegible] | [illegible] | [illegible] |
| N-NITROSODI-N-PROPYLAMINE | [illegible] | [illegible] | [illegible] | [illegible] | . | . | [illegible] | [illegible] | [illegible] | [illegible] |
| HEXACHLOROCYCLOPENTADIENE | [illegible] | <0.5 | <0.5 | <0.5 | . | . | <0.5 | [illegible] | [illegible] | [illegible] |
| 2-CHLORONAPHTHALENE | [illegible] | [illegible] | [illegible] | [illegible] | . | . | [illegible] | [illegible] | [illegible] | [illegible] |
| ISOPHORONE | [illegible] | [illegible] | [illegible] | [illegible] | . | . | [illegible] | [illegible] | [illegible] | [illegible] |
| ACENAPHTHYLENE | [illegible] | [illegible] | [illegible] | [illegible] | . | . | [illegible] | [illegible] | [illegible] | [illegible] |
| ACENAPHTHENE | [illegible] | [illegible] | [illegible] | [illegible] | . | . | [illegible] | [illegible] | [illegible] | [illegible] |
| DIMETHYL PHTHALATE | [illegible] | [illegible] | [illegible] | [illegible] | . | . | [illegible] | [illegible] | [illegible] | [illegible] |
| 2,6-DINITROTOLUENE | [illegible] | [illegible] | [illegible] | [illegible] | . | . | [illegible] | [illegible] | [illegible] | [illegible] |
| FLUORENE | [illegible] | [illegible] | [illegible] | [illegible] | . | . | [illegible] | [illegible] | [illegible] | [illegible] |
| 2,4-DINITROTOLUENE | [illegible] | [illegible] | [illegible] | [illegible] | . | . | [illegible] | [illegible] | [illegible] | [illegible] |
| 1,2-DIPHENYLHYDRAZINE | [illegible] | <0.5 | [illegible] | <0.5 | . | . | <0.5 | [illegible] | [illegible] | [illegible] |
| 4-CHLOROPHENYL PHENYL ETHER | [illegible] | [illegible] | [illegible] | [illegible] | . | . | [illegible] | [illegible] | [illegible] | [illegible] |
| DIETHYL PHTHALATE | [illegible] | [illegible] | [illegible] | [illegible] | . | . | [illegible] | [illegible] | [illegible] | [illegible] |
| N-NITROSODIPHENYLAMINE | [illegible] | [illegible] | [illegible] | [illegible] | . | . | [illegible] | [illegible] | [illegible] | [illegible] |
| HEXACHLOROBENZENE | [illegible] | [illegible] | [illegible] | [illegible] | . | . | [illegible] | [illegible] | [illegible] | [illegible] |
| 4-BROMOPHENYL PHENYL ETHER | [illegible] | [illegible] | [illegible] | [illegible] | . | . | [illegible] | [illegible] | [illegible] | [illegible] |
| PHENANTHRENE/ANTHRACENE | [illegible] | [illegible] | [illegible] | [illegible] | . | . | [illegible] | [illegible] | [illegible] | [illegible] |
| DI-N-BUTYL PHTHALATE | [illegible] | [illegible] | [illegible] | [illegible] | . | . | [illegible] | [illegible] | [illegible] | [illegible] |
| FLUORANTHENE | [illegible] | [illegible] | [illegible] | [illegible] | . | . | [illegible] | [illegible] | [illegible] | [illegible] |
| PYRENE | [illegible] | [illegible] | [illegible] | [illegible] | . | . | [illegible] | [illegible] | [illegible] | [illegible] |
| BENZIDINE | [illegible] | [illegible] | [illegible] | [illegible] | . | . | [illegible] | [illegible] | [illegible] | [illegible] |
| BUTYLBENZYL PHTHALATE | [illegible] | [illegible] | [illegible] | [illegible] | . | . | [illegible] | [illegible] | [illegible] | [illegible] |
| BIS(2-ETHYLHEXYL) PHTHALATE | [illegible] | [illegible] | [illegible] | [illegible] | . | . | [illegible] | [illegible] | [illegible] | [illegible] |
| CHRYSENE/BENZO(A)ANTHRACENE | [illegible] | [illegible] | [illegible] | [illegible] | . | . | [illegible] | [illegible] | [illegible] | [illegible] |
| 3,3'-DICHLOROBENZIDINE | [illegible] | [illegible] | [illegible] | [illegible] | . | . | [illegible] | [illegible] | [illegible] | [illegible] |
| DI-N-OCTYL PHTHALATE | [illegible] | [illegible] | [illegible] | [illegible] | . | . | [illegible] | [illegible] | [illegible] | [illegible] |
| BENZO(B)/BENZO(K)FLUORANTHENES | [illegible] | [illegible] | [illegible] | [illegible] | . | . | [illegible] | [illegible] | [illegible] | [illegible] |
| BENZO(A)PYRENE | [illegible] | [illegible] | [illegible] | [illegible] | . | . | [illegible] | [illegible] | [illegible] | [illegible] |
| INDENO(1,2,3-CD)PYRENE | [illegible] | [illegible] | [illegible] | [illegible] | . | . | [illegible] | [illegible] | [illegible] | [illegible] |
| DIBENZO(A,H)ANTHRACENE | [illegible] | [illegible] | [illegible] | [illegible] | . | . | [illegible] | [illegible] | [illegible] | [illegible] |
| BENZO(GHI)PERYLENE | 12.03 | 14 | 15 | 8.6 | . | . | 12.5333 | 3.44 | 27.5 | 104 |

## TABLE III-6. BASE/NEUTRAL EXTRACTABLE SEMIVOLATILE ORGANICS DATA—INK PIGMENT WASTE (INTERLABORATORY), SPIKE LEVEL II

| COMPOUND | ADDED | NO_1 | NO_2 | NO_3 | NO_4 | NO_5 | MEAN | S | RSD | RECOVERY |
|---|---|---|---|---|---|---|---|---|---|---|
| 1,3-DICHLOROBENZENE | 8.54 | 6.2 | 9.1 | 9.3 | 11 | 14 | 9.92 | 2.86 | 28.8 | 116 |
| 1,4-DICHLOROBENZENE | 54.61 | 44 | 58 | 62 | 67 | 83 | 62.2 | 13.12 | 21.1 | 51 |
| 1,2-DICHLOROBENZENE | 85.22 | 189 | 263 | 227 | 260 | 272 | 242.2 | 34.27 | 14.2 | 237 |
| HEXACHLOROETHANE | 13.02 | 18 | 29 | 23 | 19 | 20 | 21.8 | 4.44 | 20.4 | 218 |
| BIS(2-CHLOROETHYL) ETHER | 10 | 5.7 | 10 | 7.5 | 6.9 | 6.3 | 7.28 | 1.66 | 22.8 | 73 |
| BIS(2-CHLOROISOPROPYL) ETHER | 10.02 | 8.1 | 12 | 11 | 6.2 | 3.9 | 8.24 | 3.35 | 40.6 | 82 |
| HEXACHLOROBUTADIENE | 10.23 | 8 | 14 | 12 | 12 | 14 | 12 | 2.45 | 20.4 | 117 |
| NITROBENZENE | 50.13 | 13 | 22 | 20 | 19 | 14 | 17.6 | 3.91 | 22.2 | 35 |
| NAPHTHALENE | 160.51 | 169 | 202 | 173 | 168 | 193 | 181 | 15.51 | 8.6 | 56 |
| 1,2,4-TRICHLOROBENZENE | 11 | 17 | 24 | 21 | 21 | 30 | 22.6 | 4.83 | 21.4 | 205 |
| BIS(2-CHLOROETHOXY)METHANE | 40.34 | 39 | 56 | 45 | 41 | 37 | 43.6 | 7.54 | 17.3 | 72 |
| N-NITROSODI-N-PROPYLAMINE | 10.02 | 2.7 | 3.9 | 2.2 | 1.8 | 1.2 | 2.36 | 1.02 | 43.3 | 24 |
| HEXACHLOROCYCLOPENTADIENE | 10.12 | 1.5 | 1.8 | 1.4 | 1 | 0.29 | 1.198 | 0.58 | 48.6 | 12 |
| 2-CHLORONAPHTHALENE | 9.6 | 5.6 | 8.6 | 5.4 | 7.7 | 8.3 | 7.12 | 1.52 | 21.3 | 74 |
| ISOPHORONE | 10.08 | 21 | 25 | 20 | 23 | 17 | 21.2 | 3.03 | 14.3 | 210 |
| ACENAPHTHYLENE | 110.83 | 92 | 126 | 96 | 103 | 113 | 106 | 13.73 | 13.0 | 80 |
| ACENAPHTHENE | 85.56 | 182 | 249 | 202 | 202 | 230 | 213 | 26.40 | 12.4 | 236 |
| DIMETHYL PHTHALATE | 35.22 | 24 | 33 | 29 | 27 | 34 | 29.4 | 4.16 | 14.1 | 76 |
| 2,6-DINITROTOLUENE | 10.05 | 34 | 42 | 48 | 40 | 55 | 43.8 | 8.01 | 18.3 | 436 |
| FLUORENE | 60.24 | 59 | 81 | 68 | 68 | 80 | 71.2 | 9.26 | 13.0 | 82 |
| 2,4-DINITROTOLUENE | 60.96 | 41 | 56 | 50 | 52 | 64 | 52.6 | 8.41 | 16.0 | 86 |
| 1,2-DIPHENYLHYDRAZINE | 10.02 | 14 | 17 | 13 | 14 | 9.8 | 13.56 | 2.58 | 19.0 | 135 |
| 4-CHLOROPHENYL PHENYL ETHER | 10.24 | 6.6 | 9.9 | 9.7 | 9.6 | 9.1 | 8.98 | 1.36 | 15.2 | 89 |
| DIETHYL PHTHALATE | 10.11 | 7.2 | 10 | 11 | 13 | 8.5 | 9.94 | 2.24 | 22.5 | 64 |
| N-NITROSODIPHENYLAMINE | 10.11 | 12 | 15 | 12 | (5.3) | 12 | 12.75 | 1.50 | 11.8 | 126 |
| HEXACHLOROBENZENE | 8.07 | 5.1 | 7.4 | 5.9 | 5.9 | 6 | 6.06 | 0.83 | 13.7 | 75 |
| 4-BROMOPHENYL PHENYL ETHER | 10.33 | 6.5 | 11 | 8.5 | 1.8 | 8.5 | 7.26 | 3.44 | 47.4 | 70 |
| PHENANTHRENE/ANTHRACENE | 206.68 | 72 | 95 | 73 | 82 | 79 | 80.2 | 9.26 | 11.5 | 11 |
| DI-N-BUTYL PHTHALATE | 10.05 | 6.4 | 5.7 | 5.3 | 6.7 | 7.9 | 6.4 | 1.00 | 15.7 | 64 |
| FLUORANTHENE | 112.44 | 97 | 112 | 105 | 118 | 108 | 108 | 7.84 | 7.3 | 86 |
| PYRENE | 116.04 | 78 | 76 | 84 | 89 | 79 | 81.2 | 5.26 | 6.5 | 64 |
| BENZIDINE | 10.34 | 0.5 | 1.1 | 0.91 | 1.1 | 0.8 | 0.882 | 0.25 | 28.3 | 9 |
| BUTYLBENZYL PHTHALATE | 10.09 | 8.8 | (4.0) | 7.5 | 8.6 | 7.1 | 8 | 0.83 | 10.4 | 79 |
| BIS(2-ETHYLHEXYL) PHTHALATE | 161.16 | 64 | 68 | 69 | 57 | (23) | 64.5 | 5.45 | 8.4 | 32 |
| CHRYSENE/BENZ(A)ANTHRACENE | 63.48 | 47 | 68 | 71 | 74 | 52 | 62.4 | 12.10 | 19.4 | 92 |
| 3,3'-DICHLOROBENZIDINE | 10.04 | 1 | 14 | 12 | 10 | 4.3 | 8.26 | 5.44 | 65.9 | 82 |
| DI-N-OCTYL PHTHALATE | 10.13 | 13 | 11 | 8.6 | 6.5 | 16 | 11.02 | 3.71 | 33.7 | 109 |
| BENZO(B)/BENZO(K)FLUORANTHENES | 131.59 | 55 | 76 | 69 | 78 | 66 | 68.8 | 9.15 | 13.3 | 49 |
| BENZO(A)PYRENE | 61.88 | 47 | 96 | 75 | 94 | 75 | 77.4 | 19.73 | 25.5 | 116 |
| INDENO(1,2,3-CD)PYRENE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| DIBENZO(AH)ANTHRACENE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| BENZO(GHI)PERYLENE | 60.16 | 52 | 111 | 95 | 89 | 84 | 86.2 | 21.65 | 25.1 | 143 |

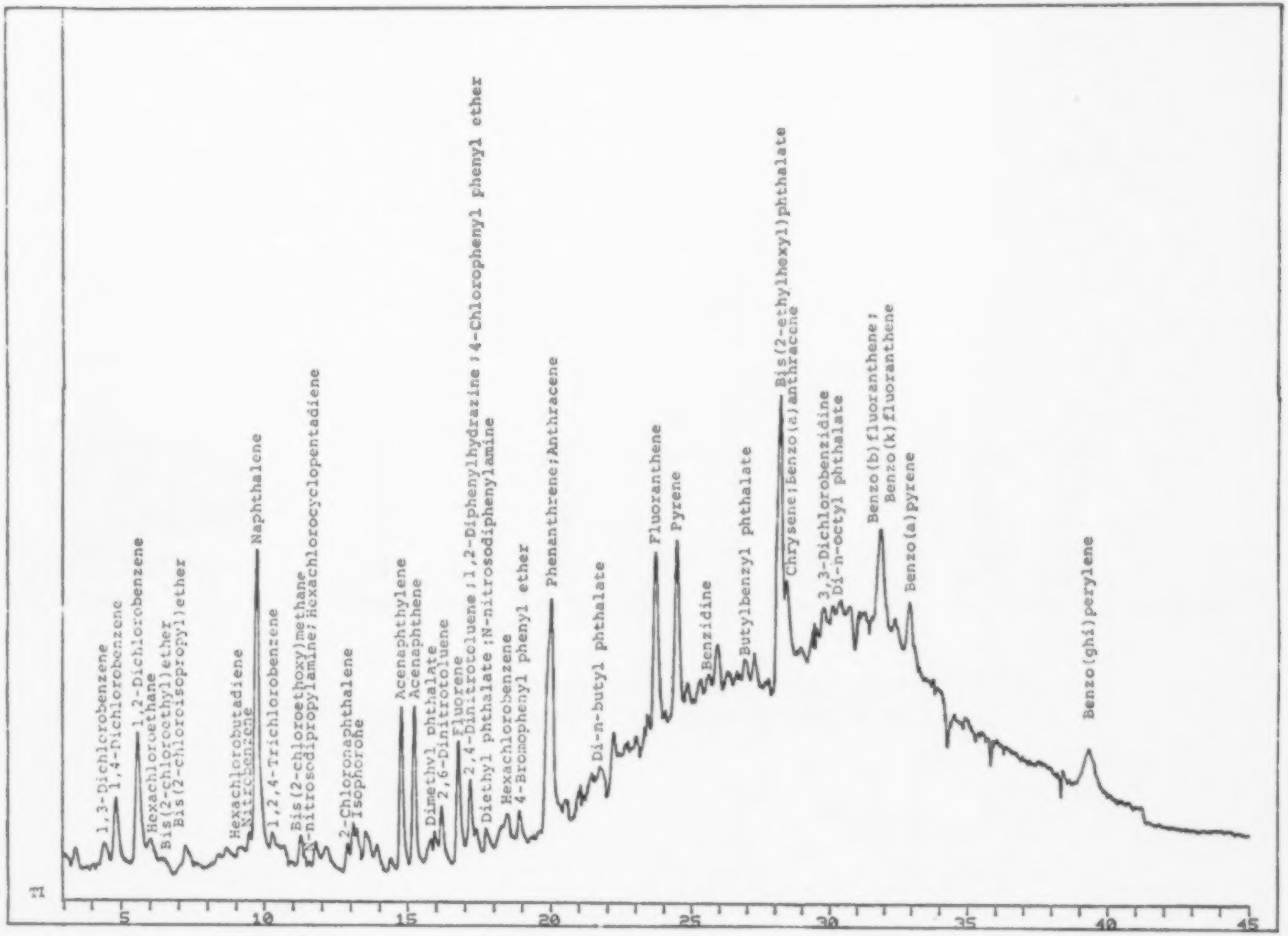

Figure III-4. GC/MS chromatogram of base/neutral extractables—Ink Pigment Waste (Interlaboratory), spiked

## TABLE III-7. ACID EXTRACTABLE SEMIVOLATILE ORGANICS DATA—INK PIGMENT WASTE (INTERLABORATORY), SPIKE LEVEL 0

| COMPOUND | ADDED | NO_1 | NO_2 | NO_3 | NO_4 | NO_5 | MEAN | S | RSD | RECOVERY |
|---|---|---|---|---|---|---|---|---|---|---|
| 2-CHLUROPHENOL | 0 | 2.5 | 4.3 | 6 | 1.8 | 3.5 | 3.62 | 1.64 | 45.2 | . |
| 2-NITROPHENOL | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| PHENOL | 0 | 77 | 73 | 59 | 66 | 67 | 68.4 | 6.91 | 10.1 | . |
| 2,4-DIMETHYLPHENUL | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| 2,4-DICHLOROPHENOL | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| 2,4,6-TRICHLOROPHENOL | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| 4-CHLORO-3-METHYLPHENOL | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| 2,4-DINITROPHENOL | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| 4,6-DINITRO-O-CRESOL | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| PENTACHLORGPHENOL | 0 | 1.2 | 2.4 | 3.3 | 2.8 | 2.7 | 2.48 | 0.79 | 31.7 | . |
| 4-NITROPHENOL | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |

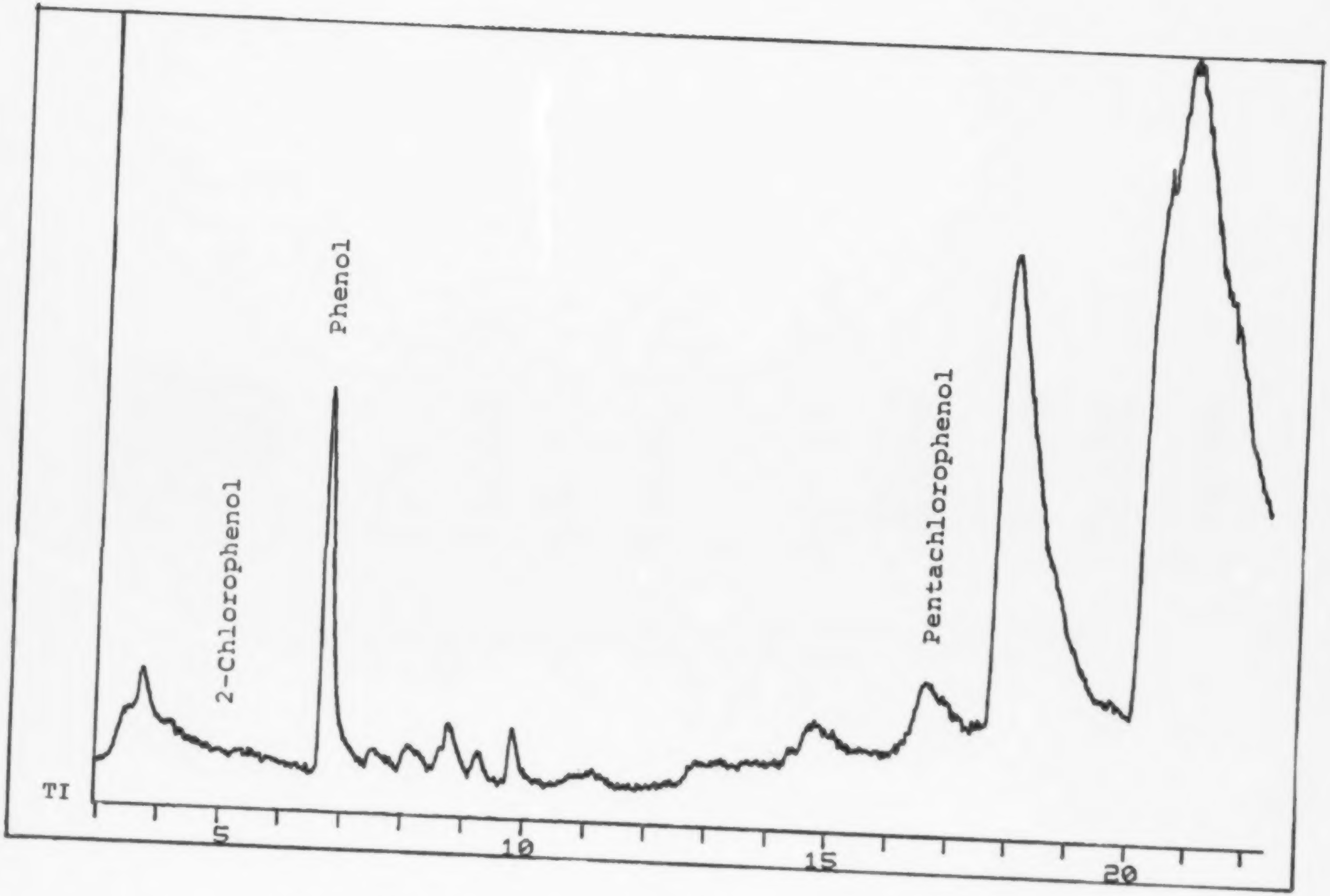

Figure III-5. GC/MS chromatogram of acid extractables — Ink Pigment Waste (Interlaboratory), unspiked

## TABLE III-8. ACID EXTRACTABLE SEMIVOLATILE ORGANICS DATA— INK PIGMENT WASTE (INTERLABORATORY), SPIKE LEVEL I

| COMPOUND | ADDED | NO_1 | NO_2 | NO_3 | NO_4 | NO_5 | MEAN | S | RSD | RECOVERY |
|---|---|---|---|---|---|---|---|---|---|---|
| 2-CHLOROPHENOL | 6.08 | 6.2 | 8.4 | 11 | . | . | 8.53333 | 2.40 | 28.2 | 81 |
| 2-NITROPHENOL | 2.01 | 1.7 | 2.5 | 3.5 | . | . | 2.56667 | 0.90 | 35.1 | 128 |
| PHENOL | 26.25 | 83 | 96 | 90 | . | . | 89.6667 | 6.51 | 7.3 | 81 |
| 2,4-DIMETHYLPHENOL | 6.27 | 2.8 | 3.3 | 5.2 | . | . | 3.76667 | 1.27 | 33.6 | 60 |
| 2,4-DICHLOROPHENOL | 2.02 | 1.5 | 4 | 3.1 | . | . | 2.86667 | 1.27 | 44.2 | 142 |
| 2,4,6-TRICHLOROPHENOL | 2.01 | 2.3 | 2.3 | 2.7 | . | . | 2.43333 | 0.23 | 9.5 | 121 |
| 4-CHLORO-3-METHYLPHENOL | 2 | 1.6 | 1.9 | 2.6 | . | . | 2.03333 | 0.51 | 25.2 | 102 |
| 2,4-DINITROPHENOL | 2 | 1.5 | 2 | 1.2 | . | . | 1.56667 | 0.40 | 25.8 | 78 |
| 4,6-DINITRO-O-CRESOL | 2.01 | 0.95 | 4.8 | 2.2 | . | . | 2.65 | 1.96 | 74.1 | 132 |
| PENTACHLOROPHENOL | 17.2 | 33 | 36 | 58 | . | . | 42.3333 | 13.65 | 32.2 | 232 |
| 4-NITROPHENOL | 14.07 | 13 | 12 | 15 | . | . | 13.3333 | 1.53 | 11.5 | 95 |

## TABLE III-9. ACID EXTRACTABLE SEMIVOLATILE ORGANICS DATA— INK PIGMENT WASTE (INTERLABORATORY), SPIKE LEVEL II

| COMPOUND | ADDED | NO_1 | NO_2 | NO_3 | NO_4 | NO_5 | MEAN | S | RSD | RECOVERY |
|---|---|---|---|---|---|---|---|---|---|---|
| 2-CHLORGPHENOL | 30.38 | 48 | 40 | 51 | 41 | 51 | 46.2 | 5.36 | 11.6 | 140 |
| 2-NITROPHENCL | 10.35 | 14 | 11 | 11 | 13 | 13 | 11.8 | 1.64 | 13.9 | 117 |
| PHENOL | 131.25 | 191 | 172 | 181 | 172 | 229 | 189 | 23.70 | 12.5 | 92 |
| 2,4-DIMETHYLPHENOL | 31.34 | 17 | 17 | 21 | 13 | 12 | 16 | 3.61 | 22.5 | 51 |
| 2,4-DICHLOROPHENOL | 10.11 | 18 | 12 | 15 | 10 | 11 | 13.2 | 3.27 | 24.8 | 131 |
| 2,4,6-TRICHLORGPHENOL | 10.06 | (19) | 10 | 12 | 10 | 12 | 11 | 1.15 | 10.5 | 109 |
| 4-CHLORO-3-METHYLPHENOL | 10.02 | 18 | 10 | 15 | 11 | 13 | 13.4 | 3.21 | 24.0 | 134 |
| 2,4-DINITROPHENOL | 10 | 15 | 9 | 13 | 16 | 17 | 14 | 3.16 | 22.6 | 140 |
| 4,6-DINITRO-O-CRESOL | 10.04 | 13 | 8.5 | 12 | 11 | 12 | 11.3 | 1.72 | 15.2 | 113 |
| PENTACHLOROPHENOL | 86.02 | 84 | 73 | 58 | 49 | 62 | 65.2 | 13.59 | 20.8 | 73 |
| 4-NITROPHENOL | 70.37 | 67 | 64 | 106 | 82 | 74 | 78.6 | 16.82 | 21.4 | 112 |

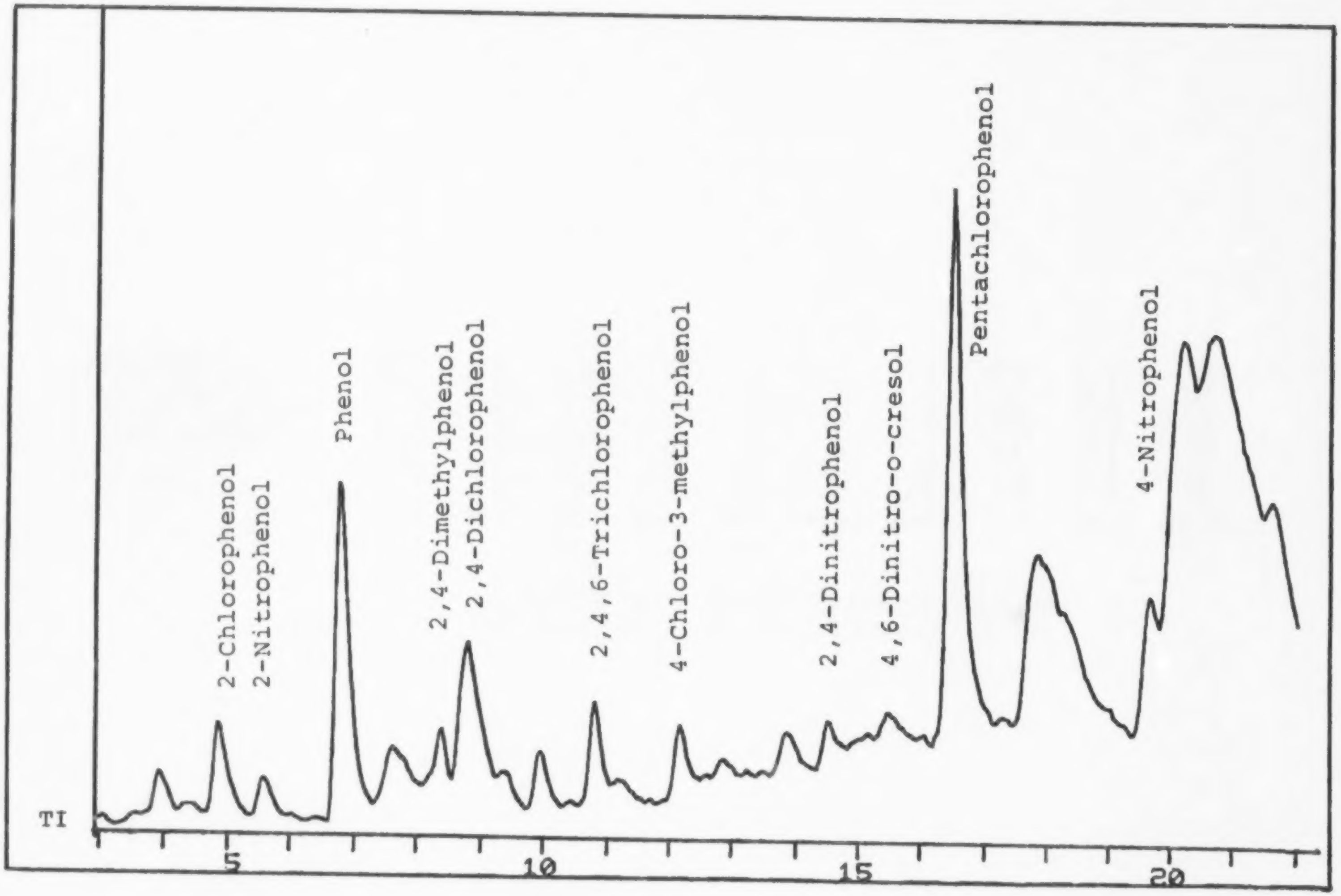

Figure III-6. GC/MS chromatogram of acid extractables—Ink Pigment Waste (Interlaboratory), spiked

TABLE III-10. METALS DATA—
INK PIGMENT WASTE (INTERLABORATORY), SPIKE LEVEL 0

| ELEMENT | ADDED | NO_1 | NO_2 | NO_3 | NO_4 | NO_5 | MEAN | S | RSD | RECOVERY |
|---|---|---|---|---|---|---|---|---|---|---|
| BERYLLIUM | 0 | <0.2 | <0.2 | <0.2 | <0.2 | <0.2 | <0.2 | Z | . | . |
| CADMIUM | 0 | <0.2 | <0.2 | <0.2 | <0.2 | <0.2 | <0.2 | Z | . | . |
| CHROMIUM | 0 | 57 | 56 | 56 | 53 | 55 | 55.4 | 1.52 | 2.7 | . |
| COPPER | 0 | 9.4 | 9.2 | 9.9 | 10 | 9.5 | 9.6 | 0.34 | 3.5 | . |
| LEAD | 0 | 242 | 237 | 241 | 238 | 245 | 240.6 | 3.21 | 1.3 | . |
| NICKEL | 0 | 1.5 | 2 | 1.7 | 1.4 | 2.1 | 1.74 | 0.30 | 17.5 | . |
| SILVER | 0 | <0.2 | <0.2 | <0.2 | <0.2 | <0.2 | <0.2 | Z | . | . |
| THALLIUM | 0 | <1 | <1 | <1 | <1 | <1 | <1 | Z | . | . |
| ZINC | 0 | 26 | 26 | 27 | 26 | 26 | 26.2 | 0.45 | 1.7 | . |

TABLE III-11. METALS DATA—
INK PIGMENT WASTE (INTERLABORATORY), SPIKE LEVEL I

| ELEMENT | ADDED | NO_1 | NO_2 | NO_3 | NO_4 | NO_5 | MEAN | S | RSD | RECOVERY |
|---|---|---|---|---|---|---|---|---|---|---|
| BERYLLIUM | 14 | 16 | 14 | 14 | 14 | 14 | 14.4 | 0.89 | 6.2 | 101 |
| CADMIUM | 14 | 14 | 14 | 14 | 14 | 14 | 14 | 0.00 | 0.0 | 99 |
| CHRCMIUM | 43 | 108 | 102 | 101 | 100 | 100 | 102.2 | 3.35 | 3.3 | 109 |
| COPPER | 29 | 41 | 38 | 37 | 37 | 35 | 37.6 | 2.19 | 5.8 | 97 |
| LEAD | 429 | 731 | 675 | 699 | 703 | 662 | 694 | 26.74 | 3.9 | 106 |
| NICKEL | 14 | 17 | 16 | 15 | 15 | 15 | 15.6 | 0.89 | 5.7 | 99 |
| SILVER | 14 | 1 | 0.8 | 0.7 | 0.5 | 0.7 | 0.74 | 0.18 | 24.5 | 4 |
| THALLIUM | 72 | 78 | 71 | 71 | 70 | 68 | 71.6 | 3.78 | 5.3 | 98 |
| ZINC | 29 | 68 | 61 | 63 | 61 | 62 | 63 | 2.92 | 4.6 | 127 |

## B. Organic Still Bottoms (Interlaboratory)

## TABLE III-12. PURGEABLE ORGANICS DATA— ORGANIC STILL BOTTOMS (INTERLABORATORY), SPIKE LEVEL 0

| COMPOUND | ADDED | NO_1 | NO_2 | NO_3 | NO_4 | NO_5 | MEAN | S | RSD | RECOVERY |
|---|---|---|---|---|---|---|---|---|---|---|
| METHYLENE CHLORIDE | 0 | 15 | 24 | 33 | 95 | 107 | 54.8 | 42.86 | 78.2 | . |
| TRICHLOROFLUOROMETHANE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| 1,1-DICHLOROETHYLENE | 0 | 132 | 118 | 99 | 65 | 61 | 95 | 31.50 | 33.2 | . |
| 1,1-DICHLOROETHANE | 0 | 2680 | 2810 | 2050 | 1830 | 1470 | 2168 | 567.82 | 26.2 | . |
| TRANS-1,2-DICHLOROETHYLENE | 0 | 974 | 1070 | 766 | 671 | 540 | 804.2 | 217.07 | 27.0 | . |
| CHLOROFORM | 0 | 321 | 332 | 231 | 224 | 203 | 262.2 | 59.72 | 22.8 | . |
| 1,2-DICHLOROETHANE | 0 | 1710 | 1800 | 1160 | 1370 | 1440 | 1496 | 259.87 | 17.4 | . |
| 1,1,1-TRICHLOROETHANE | 0 | 457 | 499 | 314 | 226 | 277 | 354.6 | 117.84 | 33.2 | . |
| CARBON TETRACHLORIDE | 0 | 77 | 89 | 53 | 35 | 44 | 59.6 | 22.69 | 38.1 | . |
| BROMODICHLOROMETHANE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| 1,2-DICHLOROPROPANE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| TRANS-1,3-DICHLOROPROPENE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| TRICHLOROETHYLENE | 0 | 6590 | 7860 | 5220 | 3730 | 5460 | 5772 | 1549.25 | 26.8 | . |
| DIBROMOCHLOROMETHANE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| CIS-1,3-DICHLOROPROPENE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| 1,1,2-TRICHLOROETHANE | 0 | 7700 | 8450 | 6190 | 6340 | 7240 | 7184 | 945.06 | 13.2 | . |
| BENZENE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| BROMOFORM | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| 1,1,2,2-TETRACHLOROETHENE | 0 | 20100 | 21100 | 16500 | (7710) | 20800 | 19625 | 2125.05 | 10.8 | . |
| 1,1,2,2-TETRACHLOROETHANE | 0 | 18900 | 38900 | 14500 | 6890 | 17400 | 19318 | 11884.62 | 61.5 | . |
| TOLUENE | 0 | 30 | 35 | 25 | 20 | 34 | 28.8 | 6.30 | 21.9 | . |
| CHLOROBENZENE | 0 | 15 | 14 | 13 | (21) | 14 | 14 | 0.82 | 5.8 | . |
| ETHYLBENZENE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |

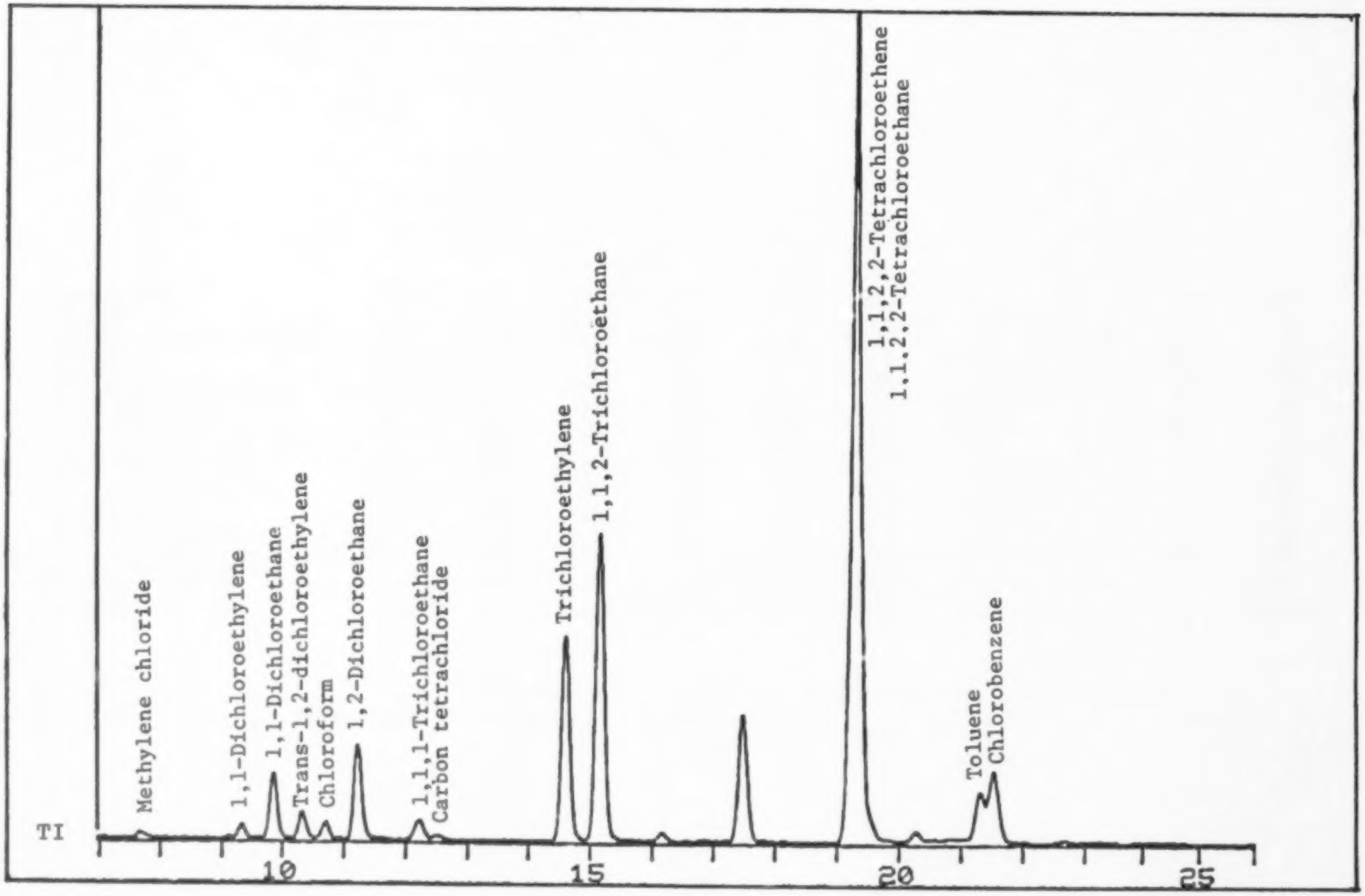

Figure III-7. GC/MS chromatogram of purgeable organics by purge and trap—Organic Still Bottoms (Interlaboratory), unspiked

TABLE III-13. PURGEABLE ORGANICS DATA—
ORGANIC STILL BOTTOMS (INTERLABORATORY), SPIKE LEVEL I

| COMPOUND | ADDED | NO_1 | NO_2 | NO_3 | NO_4 | NO_5 | MEAN | S | RSD | RECOVERY |
|---|---|---|---|---|---|---|---|---|---|---|
| METHYLENE CHLORIDE | 1990 | 2400 | 2030 | 2150 | 1820 | 1860 | 2052 | 235.52 | 11.5 | 100 |
| TRICHLOROFLUOROMETHANE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | * | * |
| 1,1-DICHLOROETHYLENE | 1990 | 2660 | 2120 | 2180 | 1710 | 1871 | 2108.2 | 362.15 | 17.2 | 101 |
| 1,1-DICHLOROETHANE | 2000 | 5250 | 4150 | 4200 | 3610 | 3230 | 4088 | 763.43 | 18.7 | 96 |
| TRANS-1,2-DICHLOROETHYLENE | 2010 | 3200 | 2840 | 2660 | 2280 | 2140 | 2624 | 427.88 | 16.3 | 91 |
| CHLOROFORM | 2000 | 2740 | 2860 | 2290 | 2020 | 2030 | 2388 | 393.66 | 16.5 | 136 |
| 1,2-DICHLOROETHANE | 2010 | 4070 | 3500 | 3940 | 2580 | 3050 | 3428 | 620.78 | 18.1 | 96 |
| 1,1,1-TRICHLOROETHANE | 2010 | 2430 | 1860 | 2040 | 1860 | 1943 | 2026 | 237.66 | 11.7 | 83 |
| CARBON TETRACHLORIDE | 2010 | 2400 | 1600 | 1930 | 1720 | 1770 | 1884 | 311.82 | 16.6 | 91 |
| BROMODICHLOROMETHANE | 1980 | 2290 | 2060 | 2170 | 2010 | 1910 | 2088 | 146.70 | 7.0 | 105 |
| 1,2-DICHLOROPROPANE | 2010 | 2980 | 2770 | 2970 | 2220 | 2730 | 2734 | 308.92 | 11.3 | 136 |
| TRANS-1,3-DICHLOROPROPENE | 2000 | 2670 | 2120 | 2270 | 1970 | 2000 | 2206 | 285.01 | 12.9 | 110 |
| TRICHLOROETHYLENE | 2000 | (12500) | 7070 | 7460 | 6670 | 7490 | 7172.5 | 385.78 | 5.4 | 70 |
| DIBROMOCHLOROMETHANE | 2010 | 2870 | 2180 | 2480 | 1970 | 2180 | 2336 | 349.47 | 15.0 | 116 |
| CIS-1,3-DICHLOROPROPENE | 1620 | 1560 | 1370 | 1720 | 1280 | 1280 | 1442 | 192.92 | 13.4 | 89 |
| 1,1,2-TRICHLOROETHANE | 2010 | 10000 | 7620 | 9140 | 8150 | 8280 | 8638 | 936.71 | 10.8 | 72 |
| BENZENE | 1990 | 2650 | 2160 | 2180 | 1850 | 1890 | 2146 | 319.58 | 14.9 | 108 |
| BROMOFORM | 2020 | 3730 | 2720 | 3300 | 3430 | 2230 | 3082 | 601.14 | 19.5 | 153 |
| 1,1,2,2-TETRACHLOROETHENE | 2010 | 21200 | 15700 | 16200 | 16500 | 19700 | 17860 | 2439.88 | 13.7 | N |
| 1,1,2,2-TETRACHLOROETHANE | 2000 | 20000 | 14900 | 15900 | 15900 | 18700 | 17080 | 2161.48 | 12.7 | N |
| TOLUENE | 2010 | 2660 | 2230 | 2440 | 2120 | 2020 | 2294 | 257.25 | 11.2 | 113 |
| CHLOROBENZENE | 2000 | 2760 | 2460 | 2890 | 2270 | 2270 | 2530 | 283.99 | 11.2 | 126 |
| ETHYLBENZENE | 1990 | 2860 | 2540 | 2380 | 2190 | 2180 | 2430 | 282.67 | 11.6 | 122 |

TABLE III-14. PURGEABLE ORGANICS DATA—
ORGANIC STILL BOTTOMS (INTERLABORATORY), SPIKE LEVEL II

| COMPOUND | ADDED | NO_1 | NO_2 | NO_3 | NO_4 | NO_5 | MEAN | S | RSD | RECOVERY |
|---|---|---|---|---|---|---|---|---|---|---|
| METHYLENE CHLORIDE | 795 | 582 | 697 | 635 | 832 | 861 | 721.4 | 121.67 | 16.9 | 84 |
| TRICHLOROFLUOROMETHANE | 800 | 635 | 846 | 589 | 1010 | 845 | 785 | 172.38 | 22.0 | 98 |
| 1,1-DICHLOROETHYLENE | 796 | 715 | 840 | 847 | 919 | 1000 | 864.2 | 105.58 | 12.2 | 97 |
| 1,1-DICHLOROETHANE | 6680 | 6570 | 6430 | 6730 | 8160 | 8650 | 7308 | 1021.82 | 14.0 | 77 |
| TRANS-1,2-DICHLOROETHYLENE | 805 | 1440 | 1080 | 1410 | 1670 | 1780 | 1476 | 270.43 | 18.3 | 83 |
| CHLOROFORM | 800 | 839 | 830 | 865 | 1010 | 1180 | 944.8 | 150.26 | 15.9 | 85 |
| 1,2-DICHLOROETHANE | 38500 | 28700 | 32100 | 26500 | 35300 | 34100 | 31340 | 3683.48 | 11.8 | 78 |
| 1,1,1-TRICHLOROETHANE | 6160 | 4280 | 5050 | 4590 | 5560 | 5980 | 5092 | 692.94 | 13.6 | 77 |
| CARBON TETRACHLORIDE | 803 | 591 | 692 | 617 | 811 | 1020 | 746.2 | 175.27 | 23.5 | 86 |
| BROMODICHLOROMETHANE | 792 | 507 | 628 | 674 | 821 | 1100 | 746 | 227.58 | 30.5 | 94 |
| 1,2-DICHLOROPROPANE | 805 | 570 | 841 | 651 | 603 | 1020 | 737 | 189.83 | 25.8 | 92 |
| TRANS-1,3-DICHLOROPROPENE | 802 | 504 | 679 | 653 | 728 | 864 | 685.6 | 130.10 | 19.0 | 85 |
| TRICHLOROETHYLENE | 40700 | 23800 | 28800 | 31600 | 41700 | 40500 | 33280 | 7677.69 | 23.1 | 68 |
| DIBROMOCHLOROMETHANE | 804 | 487 | 521 | 602 | 774 | 741 | 625 | 128.50 | 20.6 | 78 |
| CIS-1,3-DICHLOROPROPENE | 650 | 633 | 578 | 563 | 600 | 712 | 617.2 | 59.18 | 9.6 | 95 |
| 1,1,2-TRICHLOROETHANE | 26600 | 25700 | 23500 | 25400 | 28100 | 30000 | 26540 | 2532.39 | 9.5 | 73 |
| BENZENE | 797 | 0 | 0 | 0 | 0 | 0 | 0 | | | 0 |
| BROMOFORM | 810 | 561 | 799 | 735 | 696 | 907 | 739.6 | 127.84 | 17.3 | 91 |
| 1,1,2,2-TETRACHLOROETHENE | 59200 | 52300 | 50800 | 52700 | 66800 | 61500 | 56820 | 6984.77 | 12.3 | 93 |
| 1,1,2,2-TETRACHLOROETHANE | 112000 | 94400 | 97300 | 103000 | 117300 | 118000 | 105940 | 11002.64 | 10.4 | 77 |
| TOLUENE | 832 | 657 | 702 | 685 | 516 | 864 | 684.8 | 124.17 | 18.1 | 82 |
| CHLOROBENZENE | 796 | 762 | 859 | 913 | 482 | 978 | 798.8 | 193.97 | 24.3 | 99 |
| ETHYLBENZENE | 798 | 693 | 608 | 596 | 426 | 843 | 633.2 | 152.10 | 24.0 | 79 |

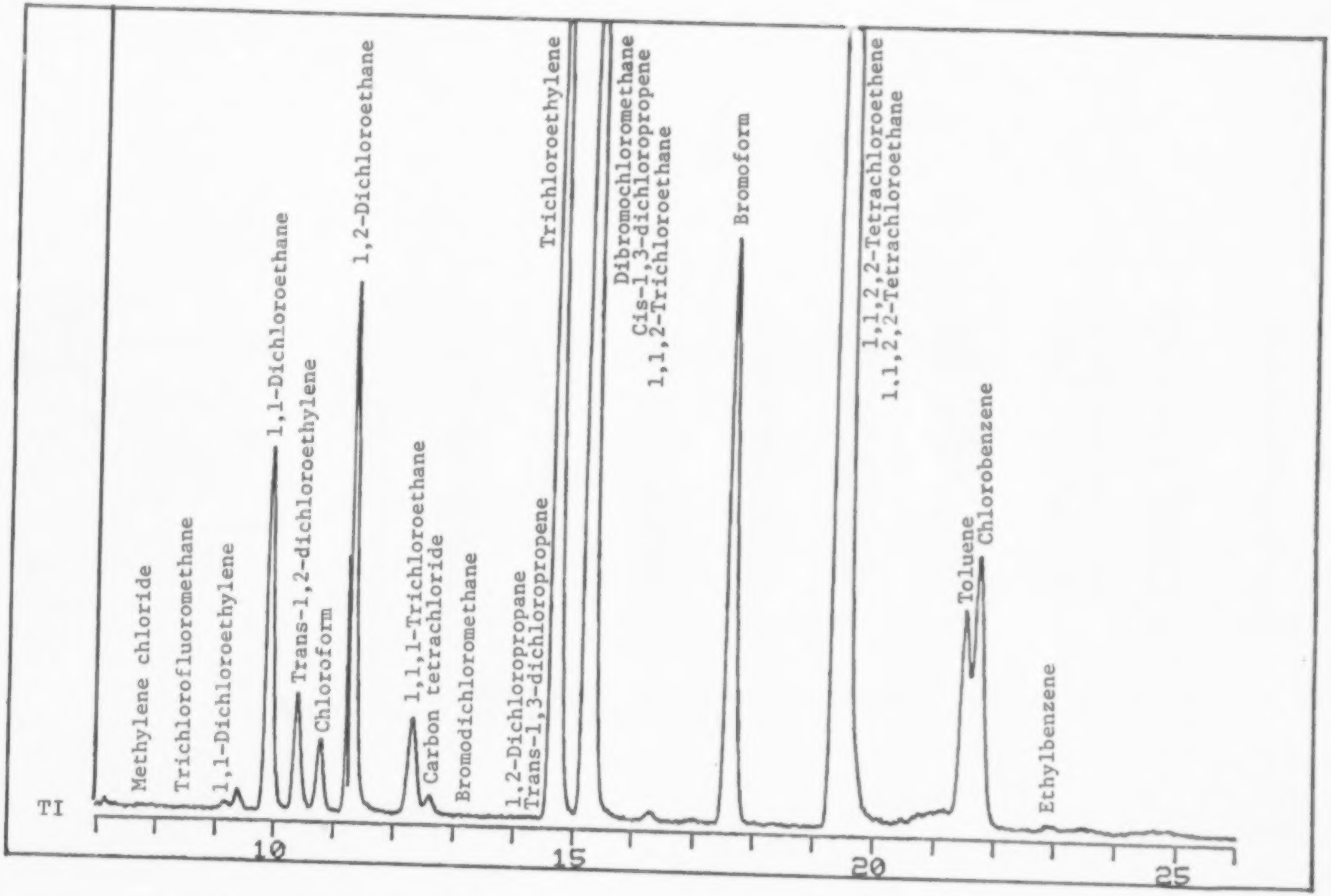

Figure III-8. GC/MS chromatogram of purgeable organics by purge and trap—Organic Still Bottoms (Interlaboratory), spiked

## TABLE III-15. BASE/NEUTRAL EXTRACTABLE SEMIVOLATILE ORGANICS DATA— ORGANIC STILL BOTTOMS (INTERLABORATORY), SPIKE LEVEL 0

| COMPOUND | ADDED | NO_1 | NO_2 | NO_3 | NO_4 | NO_5 | MEAN | S | RSD | RECOVERY |
|---|---|---|---|---|---|---|---|---|---|---|
| 1,3-DICHLOROBENZENE | 0 | 4 | 4 | 4.2 | (6.8) | 4.9 | 4.275 | 0.43 | 10.0 | . |
| 1,4-DICHLOROBENZENE | 0 | 35 | 34 | 34 | 41 | 35 | 35.8 | 2.95 | 8.2 | . |
| 1,2-DICHLOROBENZENE | 0 | 12 | 10 | 13 | 14 | 11 | 12 | 1.58 | 13.2 | . |
| HEXACHLOROETHANE | 0 | 182 | 157 | 179 | 210 | 177 | 181 | 18.96 | 10.5 | . |
| BIS(2-CHLOROETHYL) ETHER | 0 | 4.5 | 3.1 | 3.5 | 3.7 | 3.1 | 3.58 | 0.58 | 16.1 | . |
| BIS(2-CHLOROISOPROPYL) ETHER | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| HEXACHLOROBUTADIENE | 0 | 352 | 286 | 322 | 372 | 307 | 327.8 | 34.47 | 10.5 | . |
| NITROBENZENE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| NAPHTHALENE | 0 | 3.6 | 2.7 | 3.3 | 4.7 | 3.3 | 3.52 | 0.74 | 20.9 | . |
| 1,2,4-TRICHLOROBENZENE | 0 | 21 | 17 | 19 | 22 | 18 | 19.4 | 2.07 | 10.7 | . |
| BIS(2-CHLOROETHOXY)METHANE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| N-NITROSODI-N-PROPYLAMINE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| HEXACHLOROCYCLOPENTADIENE | 0 | 5.8 | 5.5 | 0 | 0 | 0 | 2.26 | 3.10 | 137.0 | . |
| 2-CHLORONAPHTHALENE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| ISOPHORONE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| ACENAPHTHYLENE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| ACENAPHTHENE | 0 | 483 | 441 | 437 | 521 | 396 | 455.6 | 47.80 | 10.5 | . |
| DIMETHYL PHTHALATE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| 2,6-DINITROTOLUENE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| FLUORENE | 0 | 0.7 | <0.5 | 0.73 | 0 | 0 | <0.386 | 0.36 | 94.1 | . |
| 2,4-DINITROTOLUENE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| 1,2-DIPHENYLHYDRAZINE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| 4-CHLOROPHENYL PHENYL ETHER | 0 | 7.4 | 6.2 | 8.4 | 10 | 8.4 | 8.08 | 1.40 | 17.4 | . |
| DIETHYL PHTHALATE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| N-NITROSODIPHENYLAMINE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| HEXACHLOROBENZENE | 0 | 132 | 115 | 136 | 161 | 128 | 134.4 | 16.83 | 12.5 | . |
| 4-BROMOPHENYL PHENYL ETHER | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| PHENANTHRENE/ANTHRACENE | 0 | 0 | 0 | <0.5 | 0.67 | <0.5 | <0.334 | 0.31 | 93.6 | . |
| DI-N-BUTYL PHTHALATE | 0 | 0.78 | 0.69 | 0.76 | 0.73 | 0.71 | 0.734 | 0.04 | 5.0 | . |
| FLUORANTHENE | 0 | 0 | 0 | 0.62 | 0.88 | 0.63 | 0.426 | 0.40 | 94.5 | . |
| PYRENE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| BENZIDINE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| BUTYLBENZYL PHTHALATE | 0 | 0.99 | 0.98 | 1.5 | 1.2 | 1.6 | 1.254 | 0.29 | 22.8 | . |
| BIS(2-ETHYLHEXYL) PHTHALATE | 0 | 3.3 | 2.9 | 2.8 | 3.7 | 2.9 | 3.12 | 0.38 | 12.1 | . |
| CHRYSENE/BENZO(A)ANTHRACENE | 0 | 0.55 | <0.5 | 0 | 1.2 | <0.5 | < 0.55 | 0.43 | 77.7 | . |
| 3,3'-DICHLOROBENZIDINE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| DI-N-OCTYL PHTHALATE | 0 | 6 | 5.9 | 6.2 | 7.3 | 8.1 | 6.7 | 0.96 | 14.4 | . |
| BENZO(B)/BENZO(K)FLUORANTHENES | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| BENZO(A)PYRENE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| INDENO(1,2,3-CD)PYRENE | 0 | (2.3) | (0.66) | 0 | 0 | 0 | 0 | Z | . | . |
| DIBENZO(AH)ANTHRACENE | 0 | (2.8) | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| BENZO(GHI)PERYLENE | 0 | (3.0) | (0.87) | 0 | 0 | 0 | 0 | Z | . | . |

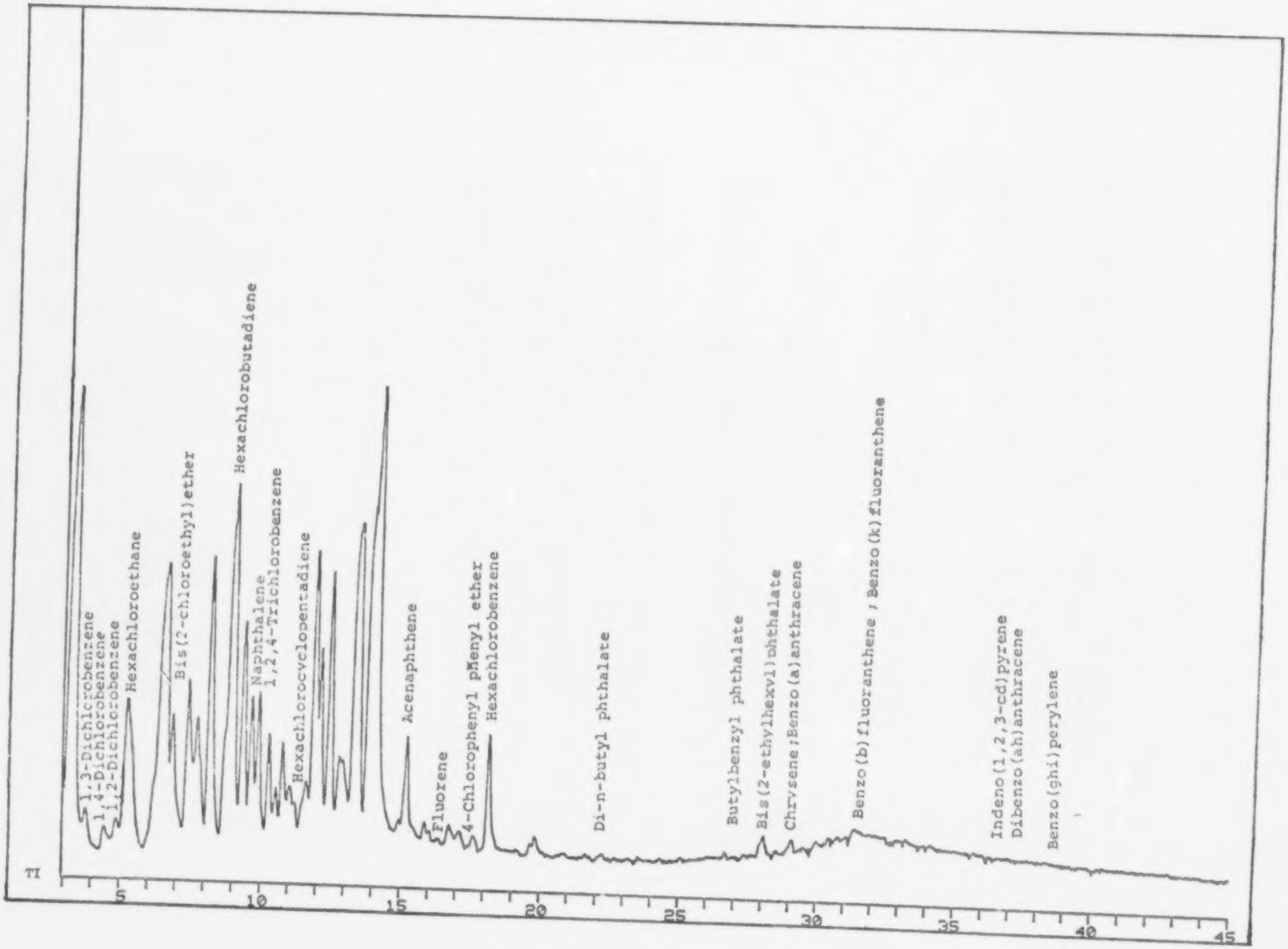

Figure III-9. GC/MS chromatogram of base/neutral extractables—Organic Still Bottoms (Interlaboratory), unspiked

TABLE III-16. BASE/NEUTRAL EXTRACTABLE SEMIVOLATILE ORGANICS DATA—
ORGANIC STILL BOTTOMS (INTERLABORATORY), SPIKE LEVEL I

| COMPOUND | ADDED | NO_1 | NO_2 | NO_3 | NO_4 | NO_5 | MEAN | S | RSD | RECOVERY |
|---|---|---|---|---|---|---|---|---|---|---|
| 1,3-DICHLOROBENZENE | 5.85 | 8.5 | 9.8 | . | . | . | 9.15 | 0.92 | 10.0 | 83 |
| 1,4-DICHLOROBENZENE | 42.82 | 37 | 62 | . | . | . | 49.5 | 17.68 | 35.7 | 32 |
| 1,2-DICHLOROBENZENE | 12.74 | 8.8 | 16 | . | . | . | 12.4 | 5.09 | 41.1 | 3 |
| HEXACHLOROETHANE | 203.35 | 144 | 248 | 151 | . | . | 181 | 58.13 | 32.1 | 0 |
| BIS(2-CHLOROETHYL) ETHER | 2 | 4.1 | 6.5 | 6.7 | . | . | 5.76667 | 1.45 | 25.1 | 109 |
| BIS(2-CHLOROISOPROPYL) ETHER | 2 | 1.1 | 1.6 | 1.8 | . | . | 1.5 | 0.36 | 24.0 | 75 |
| HEXACHLOROBUTADIENE | 360.73 | 2130 | 3200 | . | . | . | 2665 | 756.60 | 28.4 | 648 |
| NITROBENZENE | 2.01 | 3.7 | 10 | 8.7 | . | . | 7.46667 | 3.33 | 44.5 | 371 |
| NAPHTHALENE | 5.41 | 4.8 | 6.8 | . | . | . | 5.8 | 1.41 | 24.4 | 42 |
| 1,2,4-TRICHLOROBENZENE | 28.05 | 39 | 70 | . | . | . | 54.5 | 21.92 | 40.2 | 125 |
| BIS(2-CHLOROETHOXY)METHANE | 2.02 | 3.5 | 3.5 | 3.6 | . | . | 3.53333 | 0.06 | 1.6 | 175 |
| N-NITROSODI-N-PROPYLAMINE | 2 | 4.4 | 5.7 | 5.3 | . | . | 5.13333 | 0.67 | 13.0 | 257 |
| HEXACHLOROCYCLOPENTADIENE | 2.02 | 4.6 | 11 | 8.1 | . | . | 7.9 | 3.20 | 40.6 | 279 |
| 2-CHLORONAPHTHALENE | 1.92 | 1.1 | 1.6 | 1.2 | . | . | 1.3 | 0.26 | 20.4 | 68 |
| ISOPHORONE | 2.02 | 1.2 | 1.6 | 1.4 | . | . | 1.4 | 0.20 | 14.3 | 69 |
| ACENAPHTHYLENE | 2.02 | <0.5 | 0.99 | 1.6 | . | . | <1.03 | 0.55 | 53.5 | 51 |
| ACENAPHTHENE | 504.61 | 200 | 394 | . | . | . | 297 | 137.18 | 46.2 | N |
| DIMETHYL PHTHALATE | 2.01 | 1.5 | 2.2 | 2 | . | . | 1.9 | 0.36 | 19.0 | 95 |
| 2,6-DINITROTOLUENE | 2.01 | <0.5 | <0.5 | <0.5 | . | . | <0.5 | Z | . | 25 |
| FLUORENE | 2.01 | 1.7 | 2.7 | 2.1 | . | . | 2.16667 | 0.50 | 23.2 | 89 |
| 2,4-DINITROTOLUENE | 2.03 | <0.5 | <0.5 | <0.5 | . | . | <0.5 | Z | . | 25 |
| 1,2-DIPHENYLHYDRAZINE | 2 | 1.2 | 3.7 | 1.5 | . | . | 2.13333 | 1.37 | 64.0 | 107 |
| 4-CHLOROPHENYL PHENYL ETHER | 15.82 | 13 | 18 | . | . | . | 15.5 | 3.54 | 22.8 | 47 |
| DIETHYL PHTHALATE | 2.02 | 2.3 | 3.4 | 4.5 | . | . | 3.4 | 1.10 | 32.4 | 168 |
| N-NITROSODIPHENYLAMINE | 2 | <0.5 | <0.5 | <0.5 | . | . | <0.5 | Z | . | 25 |
| HEXACHLOROBENZENE | 1.61 | 141 | 246 | 297 | . | . | 228 | 79.54 | 34.9 | 5814 |
| 4-BROMOPHENYL PHENYL ETHER | 2.07 | 3 | 2.3 | 2.5 | . | . | 2.6 | 0.36 | 13.9 | 126 |
| PHENANTHRENE/ANTHRACENE | 4.04 | 3.4 | 5.1 | 5.4 | . | . | 4.63333 | 1.08 | 23.3 | 106 |
| DI-N-BUTYL PHTHALATE | 2.01 | 1.8 | 2.1 | 2.9 | . | . | 2.26667 | 0.57 | 25.1 | 76 |
| FLUORANTHENE | 2.01 | 1.6 | 2.2 | 3.5 | . | . | 2.43333 | 0.97 | 39.9 | 100 |
| PYRENE | 2.02 | 1.4 | 2 | 2.4 | . | . | 1.93333 | 0.50 | 26.0 | 96 |
| BENZIDINE | 2.01 | <0.5 | <0.5 | <0.5 | . | . | <0.5 | Z | . | 25 |
| BUTYLBENZYL PHTHALATE | 2.02 | 2.1 | 2.2 | 2.6 | . | . | 2.3 | 0.26 | 11.5 | 52 |
| BIS(2-ETHYLHEXYL) PHTHALATE | 6.62 | 5.6 | 7.7 | . | . | . | 6.65 | 1.48 | 22.3 | 53 |
| CHRYSENE/BENZO(A)ANTHRACENE | 2.02 | 1.6 | 2.2 | 2.4 | . | . | 2.06667 | 0.42 | 20.1 | 75 |
| 3,3'-DICHLOROBENZIDINE | 2.01 | <0.5 | <0.5 | <0.5 | . | . | <0.5 | Z | . | 25 |
| DI-N-OCTYL PHTHALATE | 2.03 | 2.3 | 3.2 | 3.9 | . | . | 3.13333 | 0.80 | 25.6 | N |
| BENZO(B)/BENZO(K)FLUORANTHENES | 4.42 | 3.1 | 4.6 | 4.1 | . | . | 3.93333 | 0.76 | 19.4 | 89 |
| BENZO(A)PYRENE | 2.08 | 1.7 | 1.6 | 1 | . | . | 1.43333 | 0.38 | 26.4 | 69 |
| INDENO(1,2,3-CD)PYRENE | 0 | <0.5 | <0.5 | 0.53 | . | . | <0.51 | 0.02 | 3.4 | . |
| DIBENZO(AH)ANTHRACENE | 0 | <0.5 | <0.5 | 1.1 | . | . | <0.7 | 0.35 | 49.5 | . |
| BENZO(GHI)PERYLENE | 2.02 | 2.5 | 3.5 | 5.9 | . | . | 3.96667 | 1.75 | 44.1 | 196 |

## TABLE III-17. BASE/NEUTRAL EXTRACTABLE SEMIVOLATILE ORGANICS DATA—ORGANIC STILL BOTTOMS (INTERLABORATORY), SPIKE LEVEL II

| COMPOUND | ADDED | NO_1 | NO_2 | NO_3 | NO_4 | NO_5 | MEAN | S | RSD | RECOVERY |
|---|---|---|---|---|---|---|---|---|---|---|
| 1,3-DICHLOROBENZENE | 29.23 | 41 | 73 | 61 | 58 | 21 | 50.8 | 20.20 | 39.8 | 159 |
| 1,4-DICHLOROBENZENE | 214.12 | 205 | 254 | 202 | 164 | 49 | 174.8 | 77.24 | 44.2 | 65 |
| 1,2-DICHLOROBENZENE | 63.7 | 50 | 52 | 48 | 39 | 34 | 44.6 | 7.73 | 17.3 | 51 |
| HEXACHLOROETHANE | 1016.64 | 993 | 1202 | 879 | 977 | 315 | 873.2 | 333.49 | 38.2 | 68 |
| BIS(2-CHLOROETHYL) ETHER | 10 | 11 | 17 | 11 | 12 | 11 | 12.4 | 2.61 | 21.0 | 88 |
| BIS(2-CHLOROISOPROPYL) ETHER | 10.02 | 6.1 | 9.9 | 7.8 | 7.7 | 8.3 | 7.96 | 1.36 | 17.1 | 79 |
| HEXACHLOROBUTADIENE | 1803.61 | 2420 | 2560 | 1840 | 1920 | 671 | 1882.2 | 744.79 | 39.6 | 86 |
| NITROBENZENE | 10.03 | 1.9 | 32 | 5.8 | 19 | 17 | 15.14 | 11.89 | 78.5 | 151 |
| NAPHTHALENE | 27.07 | 18 | 29 | 22 | 27 | 13 | 21.8 | 6.53 | 30.0 | 68 |
| 1,2,4-TRICHLOROBENZENE | 140.25 | 154 | 175 | 134 | 137 | (50) | 150 | 18.85 | 12.6 | 93 |
| BIS(2-CHLOROETHOXY)METHANE | 10.09 | 22 | 26 | 18 | 18 | 21 | 21 | 3.32 | 15.8 | 208 |
| N-NITROSODI-N-PROPYLAMINE | 10.02 | 3.1 | 7.1 | 5.4 | 3.4 | 3.9 | 4.58 | 1.66 | 36.3 | 46 |
| HEXACHLOROCYCLOPENTADIENE | 10.12 | 3.7 | 9.2 | 5.8 | 7.7 | 4.2 | 6.12 | 2.33 | 38.0 | 38 |
| 2-CHLORONAPHTHALENE | 9.6 | 5.8 | 6.9 | 4.7 | 5.7 | 6.4 | 5.9 | 0.83 | 14.0 | 61 |
| ISOPHORONE | 10.08 | 11 | 6.4 | 2.5 | 8.1 | 3.6 | 6.32 | 3.43 | 54.3 | 63 |
| ACENAPHTHYLENE | 10.08 | 5.9 | 9.1 | 8.5 | 4.2 | 5.9 | 6.72 | 2.03 | 30.2 | 57 |
| ACENAPHTHENE | 2523.06 | 1590 | 2120 | 1500 | 1260 | 868 | 1467.6 | 459.42 | 31.3 | 40 |
| DIMETHYL PHTHALATE | 10.07 | 9.7 | 13 | 8.9 | 8.8 | 9.7 | 10.02 | 1.72 | 17.2 | 100 |
| 2,6-DINITROTOLUENE | 10.05 | 8.7 | 10 | 6.3 | 8 | 6.1 | 7.82 | 1.65 | 21.0 | 78 |
| FLUORENE | 10.04 | 8.6 | 12 | 8.2 | 8.6 | 10 | 9.48 | 1.57 | 16.5 | 91 |
| 2,4-DINITROTOLUENE | 10.16 | 8.5 | 14 | 6.4 | 7.8 | 7.7 | 8.88 | 2.96 | 33.3 | 87 |
| 1,2-DIPHENYLHYDRAZINE | 10.02 | 17 | 16 | 10 | 12 | 12 | 13.4 | 2.97 | 22.1 | 134 |
| 4-CHLOROPHENYL PHENYL ETHER | 79.12 | 65 | 84 | 63 | 71 | (17) | 70.75 | 9.46 | 13.4 | 79 |
| DIETHYL PHTHALATE | 10.11 | 8.2 | 14 | 6.9 | 8 | 11 | 9.62 | 2.88 | 29.9 | 95 |
| N-NITROSODIPHENYLAMINE | 10 | 20 | 11 | 22 | 3.9 | 16 | 14.58 | 7.30 | 50.1 | 146 |
| HEXACHLOROBENZENE | 8.07 | 146 | (217) | 125 | 131 | 150 | 138 | 11.92 | 8.6 | 45 |
| 4-BROMOPHENYL PHENYL ETHER | 10.33 | 7.3 | 13 | 9.8 | 12 | 12 | 10.82 | 2.29 | 21.2 | 105 |
| PHENANTHRENE/ANTHRACENE | 20.18 | 8.2 | 9 | 8.7 | 8.4 | 7.8 | 8.42 | 0.46 | 5.5 | 40 |
| DI-N-BUTYL PHTHALATE | 10.05 | 10 | 12 | 8 | 9.7 | 10 | 9.94 | 1.42 | 14.3 | 92 |
| FLUORANTHENE | 10.04 | 12 | 14 | 8.6 | 8.8 | 10 | 10.68 | 2.30 | 21.5 | 102 |
| PYRENE | 10.09 | 11 | 16 | 9.4 | 8.9 | 7.5 | 10.56 | 3.29 | 31.1 | 105 |
| BENZIDINE | 10.04 | 5.3 | 9.6 | 5.2 | 2.3 | 4.3 | 5.34 | 2.67 | 50.0 | 53 |
| BUTYLBENZYL PHTHALATE | 10.09 | 11 | 13 | 8.5 | 8.5 | 8.1 | 9.82 | 2.12 | 21.6 | 85 |
| BIS(2-ETHYLHEXYL) PHTHALATE | 33.08 | 63 | 51 | 35 | 30 | 19 | 39.6 | 17.43 | 44.0 | 110 |
| CHRYSENE/BENZO(A)ANTHRACENE | 10.08 | 13 | 15 | 12 | 11 | 10 | 12.2 | 1.92 | 15.8 | 116 |
| 3,3'-DICHLOROBENZIDINE | 10.04 | <1 | (4.7) | 1.3 | 1.1 | 1 | <1.1 | 0.14 | 12.9 | 11 |
| DI-N-OCTYL PHTHALATE | 10.13 | 8 | 7.3 | 4.8 | 5.7 | 7.6 | 6.68 | 1.37 | 20.5 | N |
| BENZO(B)/BENZO(K)FLUORANTHENES | 22.11 | 12 | 14 | 9.2 | 11 | 11 | 11.44 | 1.75 | 15.3 | 52 |
| BENZO(A)PYRENE | 10.4 | 10 | 13 | 13 | 11 | 9.9 | 11.38 | 1.54 | 13.5 | 109 |
| INDENO(1,2,3-CD)PYRENE | 0 | <1 | <1 | <1 | <1 | <1 | <1 | Z | • | • |
| DIBENZO(AH)ANTHRACENE | 0 | <1 | <1 | <1 | <1 | <1 | <1 | Z | • | • |
| BENZO(GHI)PERYLENE | 10.12 | 8.7 | 14 | 9.4 | 9.4 | 11 | 10.5 | 2.13 | 20.3 | 104 |

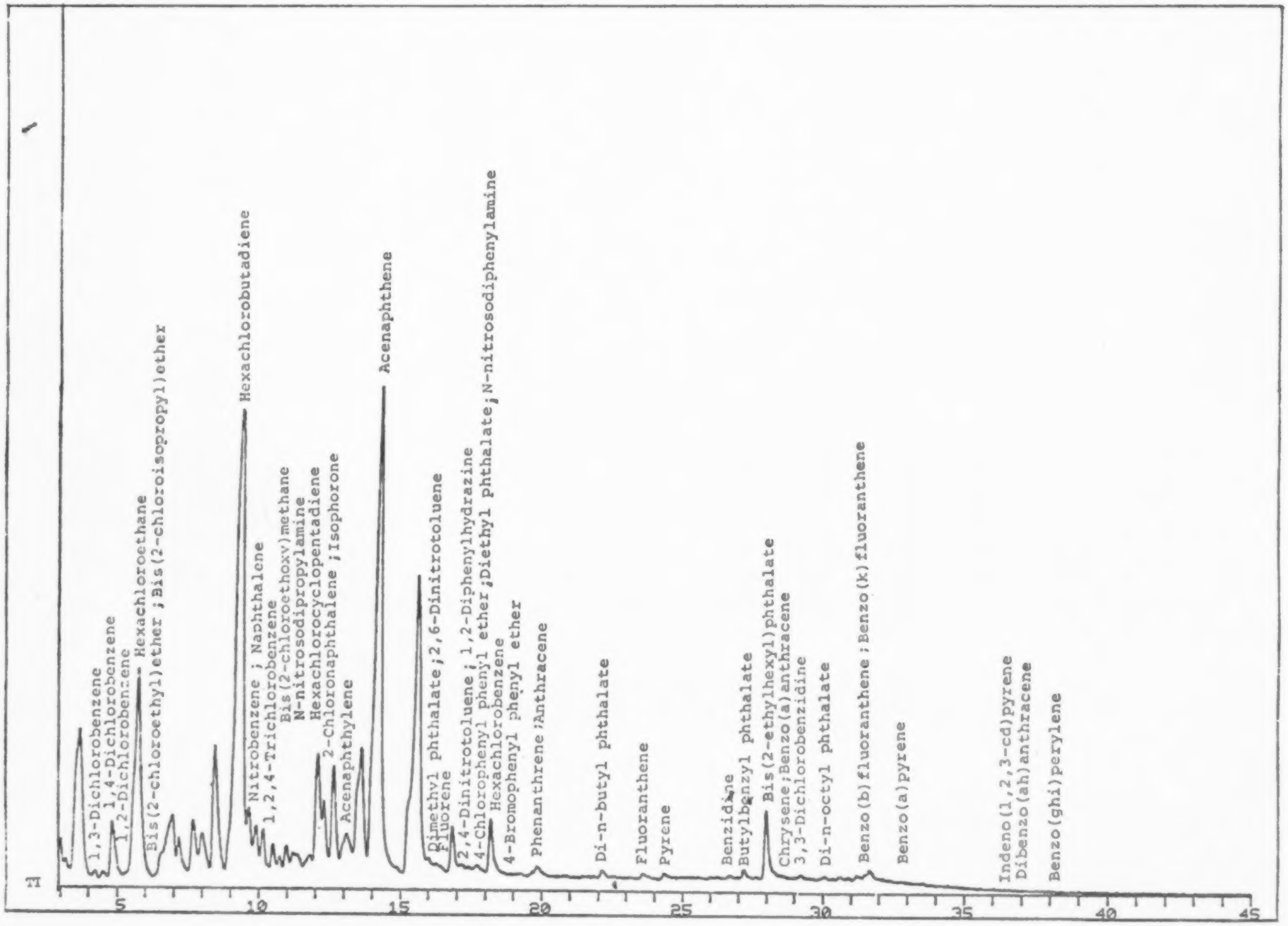

Figure III-10. GC/MS chromatogram of base/neutral extractables—Organic Still Bottoms (Interlaboratory), spiked

TABLE III-18. ACID EXTRACTABLE SEMIVOLATILE ORGANICS DATA—
ORGANIC STILL BOTTOMS (INTERLABORATORY), SPIKE LEVEL 0

| COMPOUND | ADDED | NO_1 | NO_2 | NO_3 | NO_4 | NO_5 | MEAN | S | RSD | RECOVERY |
|---|---|---|---|---|---|---|---|---|---|---|
| 2-CHLOROPHENOL | 0 | 0.93 | 0.86 | 0.44 | 0.53 | 0.54 | 0.66 | 0.22 | 33.2 | . |
| 2-NITROPHENOL | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| PHENOL | 0 | 6.6 | 7.1 | 5.6 | 4.6 | 6.2 | 6.02 | 0.97 | 16.0 | . |
| 2,4-DIMETHYLPHENOL | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| 2,4-DICHLOROPHENOL | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| 2,4,6-TRICHLOROPHENOL | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| 4-CHLORO-3-METHYLPHENOL | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| 2,4-DINITROPHENOL | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| 4,6-DINITRO-O-CRESOL | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| PENTACHLOROPHENOL | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| 4-NITROPHENOL | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |

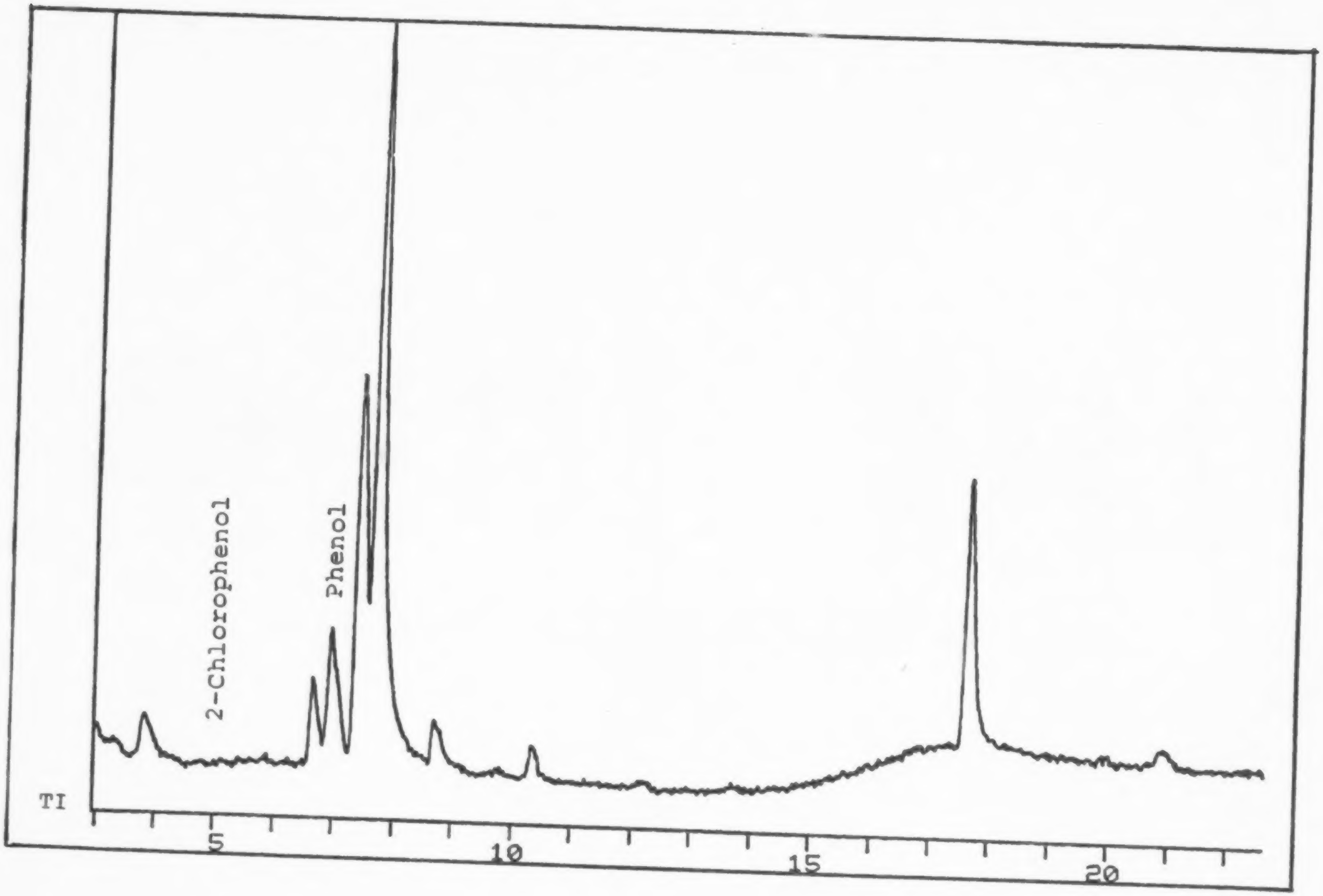

Figure III-11. GC/MS chromatogram of acid extractables—
Organic Still Bottoms (Interlaboratory), unspiked

TABLE III-19. ACID EXTRACTABLE SEMIVOLATILE ORGANICS DATA—
ORGANIC STILL BOTTOMS (INTERLABORATORY), SPIKE LEVEL I

| COMPOUND | ADDED | NO_1 | NO_2 | NO_3 | NO_4 | NO_5 | MEAN | S | RSD | RECOVERY |
|---|---|---|---|---|---|---|---|---|---|---|
| 2-CHLOROPHENOL | 2.01 | 2.4 | 1.7 | 2.4 | . | . | 2.16667 | 0.40 | 18.7 | 75 |
| 2-NITROPHENOL | 2.01 | 0.55 | 0.55 | <0.5 | . | . | <.533333 | 0.03 | 5.4 | 27 |
| PHENOL | 8.56 | 12 | 12 | . | . | . | 12 | 0.00 | 0.0 | 70 |
| 2,4-DIMETHYLPHENOL | 2.01 | <0.5 | <0.5 | <0.5 | . | . | <0.5 | Z | . | 25 |
| 2,4-DICHLOROPHENOL | 2.02 | 2.3 | 1.2 | 1.6 | . | . | 1.7 | 0.56 | 32.8 | 84 |
| 2,4,6-TRICHLOROPHENOL | 2.01 | 2.4 | 1.1 | 2.5 | . | . | 2 | 0.78 | 39.1 | 100 |
| 4-CHLORO-3-METHYLPHENOL | 2 | 0.87 | 0.95 | 1 | . | . | 0.94 | 0.07 | 7.0 | 47 |
| 2,4-DINITROPHENOL | 2 | 0.59 | <0.5 | <0.5 | . | . | <0.53 | 0.05 | 9.8 | 26 |
| 4,6-DINITRO-O-CRESOL | 2.01 | 0.58 | <0.5 | <0.5 | . | . | <.526667 | 0.05 | 8.8 | 26 |
| PENTACHLOROPHENOL | 2 | 1.4 | 1.3 | 1.6 | . | . | 1.43333 | 0.15 | 10.7 | 72 |
| 4-NITROPHENOL | 2 | 0.56 | 0.56 | 0.91 | . | . | 0.676667 | 0.20 | 29.9 | 34 |

TABLE III-20. ACID EXTRACTABLE SEMIVOLATILE ORGANICS DATA—
ORGANIC STILL BOTTOMS (INTERLABORATORY), SPIKE LEVEL II

| COMPOUND | ADDED | NO_1 | NO_2 | NO_3 | NO_4 | NO_5 | MEAN | S | RSD | RECOVERY |
|---|---|---|---|---|---|---|---|---|---|---|
| 2-CHLOROPHENOL | 10.03 | 7.2 | 7.4 | 7.7 | 12 | <1 | <7.06 | 3.93 | 55.6 | 64 |
| 2-NITROPHENOL | 10.05 | <1 | <1 | <1 | <1 | <1 | <1 | Z | . | 10 |
| PHENOL | 42.82 | 35 | 41 | 48 | 51 | (1.3) | 43.75 | 7.18 | 16.4 | 88 |
| 2,4-DIMETHYLPHENOL | 10.04 | 2.2 | 1.8 | 1.5 | (8.8) | 1.4 | 1.725 | 0.36 | 20.8 | 17 |
| 2,4-DICHLOROPHENOL | 10.11 | 8.8 | 8 | 6.2 | 7.1 | 2.2 | 6.46 | 2.57 | 39.8 | 64 |
| 2,4,6-TRICHLOROPHENOL | 10.06 | 7.8 | 8.7 | 6.9 | 11 | 2.1 | 7.3 | 3.28 | 45.0 | 73 |
| 4-CHLORO-3-METHYLPHENOL | 10.02 | 6.5 | 7.5 | 9 | 9.9 | <1 | <6.78 | 3.49 | 51.4 | 68 |
| 2,4-DINITROPHENOL | 10 | <1 | <1 | <1 | <1 | <1 | <1 | Z | . | 10 |
| 4,6-DINITRO-O-CRESOL | 10.04 | <1 | <1 | <1 | <1 | <1 | <1 | Z | . | 10 |
| PENTACHLOROPHENOL | 10.02 | 4 | 5.8 | 5.5 | 6.3 | 3 | 4.92 | 1.37 | 27.9 | 49 |
| 4-NITROPHENOL | 10.02 | <1 | <1 | <1 | <1 | <1 | <1 | Z | . | 10 |

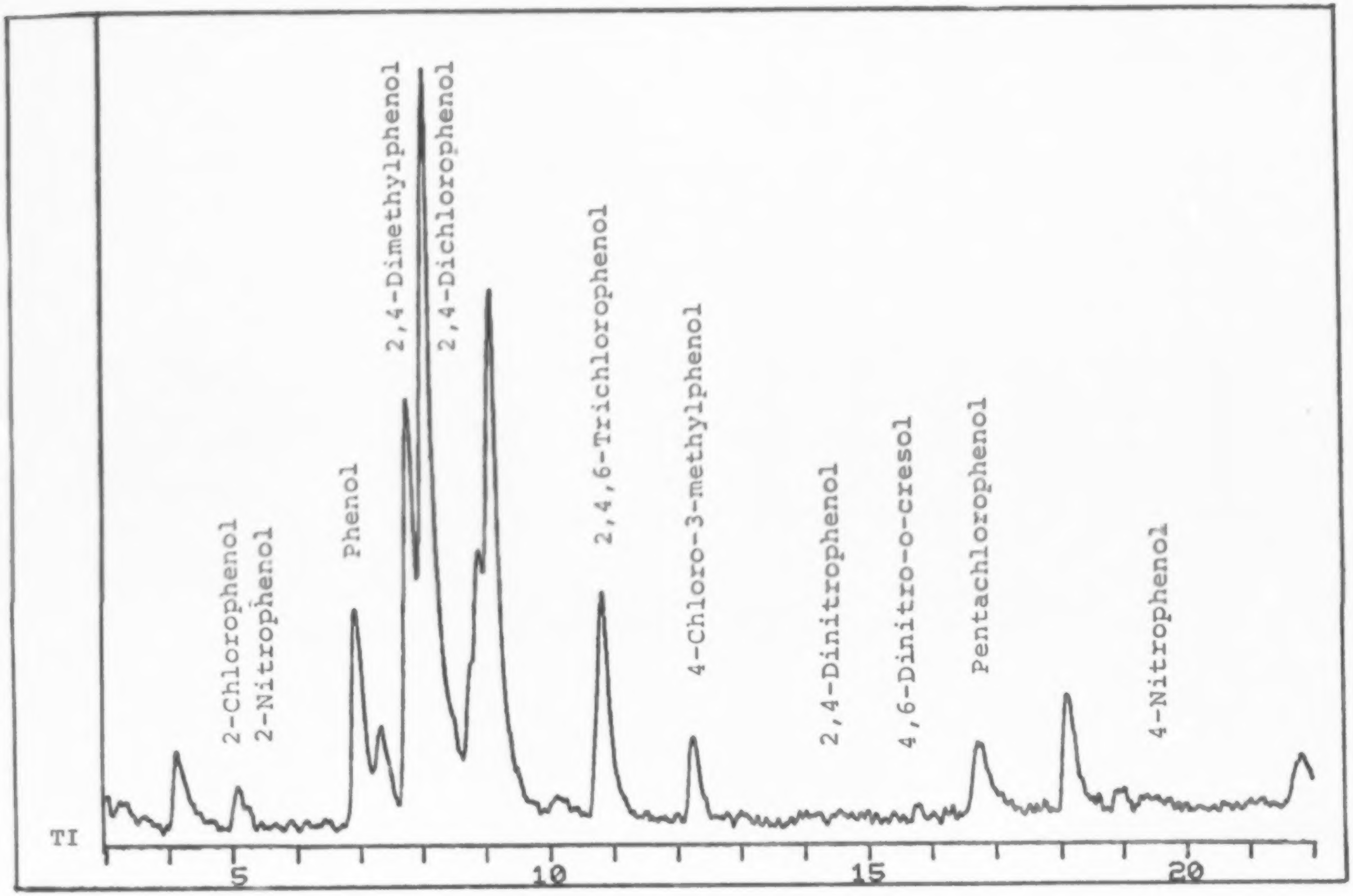

Figure III-12. GC/MS chromatogram of acid extractables—
Organic Still Bottoms (Interlaboratory), spiked

## TABLE III-21. METALS DATA— ORGANIC STILL BOTTOMS (INTERLABORATORY), SPIKE LEVEL 0

| ELEMENT | ADDED | NO_1 | NO_2 | NO_3 | NO_4 | NO_5 | MEAN | S | RSD | RECOVERY |
|---|---|---|---|---|---|---|---|---|---|---|
| BERYLLIUM | 0 | <0.2 | <0.2 | <0.2 | <0.2 | <0.2 | <0.2 | Z | . | . |
| CADMIUM | 0 | <0.2 | <0.2 | <0.2 | <0.2 | <0.2 | <0.2 | Z | . | . |
| CHROMIUM | 0 | 50 | 38 | 30 | 36 | 41 | 39 | 7.35 | 18.8 | . |
| COPPER | 0 | 885 | 821 | 919 | 902 | 893 | 884 | 37.42 | 4.2 | . |
| LEAD | 0 | 22 | 22 | 21 | 21 | 22 | 21.6 | 0.55 | 2.5 | . |
| NICKEL | 0 | 107 | 84 | 79 | 98 | 88 | 91.2 | 11.26 | 12.3 | . |
| SILVER | 0 | <0.2 | <0.2 | <0.2 | <0.2 | <0.2 | <0.2 | Z | . | . |
| THALLIUM | 0 | <1 | <1 | <1 | <1 | <1 | <1 | Z | . | . |
| ZINC | 0 | 213 | 223 | 229 | 226 | 221 | 222.4 | 6.07 | 2.7 | . |

TABLE III-22. METALS DATA—
ORGANIC STILL BOTTOMS (INTERLABORATORY), SPIKE LEVEL I

| ELEMENT | ADDED | NO_1 | NO_2 | NO_3 | NO_4 | NO_5 | MEAN | S | RSD | RECOVERY |
|---|---|---|---|---|---|---|---|---|---|---|
| BERYLLIUM | 17 | 15 | 13 | 15 | 15 | 14 | 14.4 | 0.89 | 6.2 | 84 |
| CADMIUM | 17 | 17 | 17 | 19 | 19 | 17 | 17.8 | 1.10 | 6.2 | 104 |
| CHROMIUM | 34 | 90 | 78 | 82 | 82 | 88 | 84 | 4.90 | 5.8 | 132 |
| COPPER | 850 | 1500 | 1730 | 1280 | 1360 | 1500 | 1474 | 171.41 | 11.6 | 69 |
| LEAD | 17 | 43 | 39 | 44 | 43 | 41 | 42 | 2.00 | 4.8 | 120 |
| NICKEL | 340 | 381 | 389 | 333 | 364 | 393 | 372 | 24.47 | 6.6 | 83 |
| SILVER | 17 | 3.9 | 4.4 | 3.4 | 3 | 5.1 | 3.96 | 0.83 | 20.9 | 22 |
| THALLIUM | 85 | 82 | 76 | 87 | 87 | 76 | 81.6 | 5.50 | 6.7 | 95 |
| ZINC | 85 | 289 | 309 | 309 | 311 | 299 | 303.4 | 9.32 | 3.1 | 95 |

# Appendix IV. Total Content Evaluation Data

## Legend for Verification Analyses and Quality Assurance for Total Content Validation Study Data*

| Symbols | Identification |
|---|---|
| . | Not determined. |
| < | Lower detection limit of method. |
| ( ) | Found to be a statistical outlier and not included in the calculations. |
| Z | Standard deviation was not calculated because all points in the data set were less than lower detection limit of method and/or zero. |
| N | Percent recovery was calculated to be less than zero. |

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* Concentrations are reported as microgram of compound per gram of residual waste (parts per million).

## A. Deionized, Distilled Water (Spiked Method Blank)

## TABLE IV-1. PERCENT RECOVERY OF ORGANICS FROM SPIKED METHOD BLANK

| COMPOUND | NO_1 | NO_2 | NO_3 | NO_4 | NO_5 | MEAN | S | RSD |
|---|---|---|---|---|---|---|---|---|
| 1,3-DICHLOROBENZENE | 33 | 83 | 62 | 6 | 15 | 39.8 | 32.3 | 81.05 |
| 1,4-DICHLOROBENZENE | 62 | 93 | 82 | 35 | 45 | 63.4 | 24.3 | 38.39 |
| 1,2-DICHLOROBENZENE | 86 | 111 | 180 | 128 | 85 | 118.0 | 39.1 | 33.11 |
| HEXACHLOROETHANE | 78 | 86 | 154 | 124 | 65 | 101.4 | 36.7 | 36.19 |
| BIS(2-CHLOROETHYL) ETHER | 86 | 77 | 72 | 23 | 19 | 55.4 | 31.8 | 57.46 |
| BIS(2-CHLOROISOPROPYL) ETHER | 70 | 47 | 97 | 58 | 112 | 76.8 | 27.1 | 35.27 |
| HEXACHLOROBUTADIENE | 131 | 119 | 120 | 115 | 110 | 119.0 | 7.8 | 6.54 |
| NITROBENZENE | 15 | 48 | 39 | 120 | 102 | 64.8 | 44.3 | 68.40 |
| NAPHTHALENE | 82 | 102 | 93 | 84 | 102 | 92.6 | 9.5 | 10.29 |
| 1,2,4-TRICHLOROBENZENE | 108 | 17 | 174 | 171 | 73 | 108.6 | 66.8 | 61.48 |
| BIS(2-CHLOROETHOXY)METHANE | 64 | 56 | 116 | 86 | 58 | 76.0 | 25.3 | 33.34 |
| N-NITROSODI-N-PROPYLAMINE | 70 | 78 | 42 | 53 | 82 | 65.0 | 17.0 | 26.15 |
| HEXACHLOROCYCLOPENTADIENE | 5 | 105 | 29 | 17 | 12 | 33.6 | 40.9 | 121.62 |
| 2-CHLORONAPHTHALENE | 102 | 109 | 67 | 80 | 134 | 98.4 | 26.1 | 26.49 |
| ISOPHORONE | 47 | 50 | 295 | 163 | 140 | 139.0 | 101.6 | 73.10 |
| ACENAPHTHYLENE | 100 | 97 | 88 | 75 | 114 | 94.8 | 14.5 | 15.28 |
| ACENAPHTHENE | 99 | 92 | 230 | 212 | 115 | 149.6 | 66.0 | 44.13 |
| DIMETHYL PHTHALATE | 112 | 93 | 82 | 58 | 78 | 84.6 | 19.9 | 23.49 |
| 2,6-DINITROTOLUENE | 308 | 58 | 370 | 82 | 108 | 185.2 | 143.2 | 77.32 |
| FLUORENE | 107 | 96 | 97 | 105 | 108 | 102.6 | 5.7 | 5.54 |
| 2,4-DINITROTOLUENE | 94 | 66 | 85 | 83 | 81 | 81.8 | 10.1 | 12.39 |
| 1,2-DIPHENYLHYDRAZINE | 46 | 40 | 180 | 113 | 118 | 99.4 | 57.9 | 58.24 |
| 4-CHLOROPHENYL PHENYL ETHER | 118 | 96 | 89 | 106 | 90 | 99.8 | 12.2 | 12.24 |
| DIETHYL PHTHALATE | 158 | 91 | 105 | 89 | 236 | 135.8 | 62.6 | 46.10 |
| N-NITROSODIPHENYLAMINE | 125 | 55 | 67 | 58 | 12 | 63.4 | 40.5 | 63.82 |
| HEXACHLOROBENZENE | 112 | 100 | 74 | 96 | 52 | 86.8 | 23.8 | 27.44 |
| 4-BROMOPHENYL PHENYL ETHER | 129 | 90 | 70 | 94 | 87 | 94.0 | 21.6 | 22.98 |
| PHENANTHRENE/ANTHRACENE | 92 | 89 | 32 | 35 | 100 | 69.6 | 33.2 | 47.72 |
| DI-N-BUTYL PHTHALATE | 106 | 92 | 66 | 71 | 124 | 91.8 | 24.2 | 26.33 |
| FLUORANTHENE | 93 | 91 | 85 | 96 | 104 | 93.8 | 7.0 | 7.44 |
| PYRENE | 85 | 95 | 74 | 94 | 125 | 94.6 | 19.0 | 20.07 |
| BENZIDINE | 9 | 68 | 21 | 5 | 12 | 23.0 | 25.8 | 112.33 |
| BUTYLBENZYL PHTHALATE | 88 | 44 | 88 | 69 | 81 | 74.0 | 18.5 | 24.97 |
| BIS(2-ETHYLHEXYL) PHTHALATE | 153 | 33 | 58 | 119 | 106 | 93.8 | 48.1 | 51.31 |
| CHRYSENE/BENZO(A)ANTHRACENE | 122 | 87 | 598 | 81 | 88 | 195.2 | 225.7 | 115.65 |
| 3,3'-DICHLOROBENZIDINE | 32 | 55 | 71 | 100 | 46 | 60.8 | 26.1 | 42.91 |
| DI-N-OCTYL PHTHALATE | 5 | 2 | 5 | 5 | 12 | 5.8 | 3.7 | 63.82 |
| BENZO(B)/BENZO(K)FLUORANTHENES | 131 | 157 | 37 | 42 | 69 | 87.2 | 54.0 | 61.98 |
| BENZO(A)PYRENE | 216 | 165 | 61 | 97 | 82 | 124.2 | 64.4 | 51.88 |
| INDENO(1,2,3-CD)PYRENE | 112 | . | . | . | . | 112.0 | . | . |
| DIBENZO(AH)ANTHRACENE | 129 | . | . | . | . | 129.0 | . | . |
| BENZO(GHI)PERYLENE | 98 | 132 | 78 | 100 | 42 | 90.0 | 33.1 | 36.75 |
| 2-CHLOROPHENOL | 53 | 70 | 104 | 87 | 80 | 78.8 | 19.0 | 24.14 |
| 2-NITROPHENOL | 48 | 93 | 122 | 85 | 61 | 81.8 | 28.8 | 35.26 |
| PHENOL | 36 | 63 | 121 | 107 | 44 | 74.2 | 38.0 | 51.15 |
| 2,4-DIMETHYLPHENOL | 6 | 4 | 40 | 50 | 19 | 23.8 | 20.5 | 86.13 |
| 2,4-DICHLOROPHENOL | 32 | 51 | 145 | 85 | 164 | 95.4 | 57.6 | 60.36 |
| 2,4,6-TRICHLOROPHENOL | 20 | 164 | 115 | 83 | 294 | 135.2 | 103.0 | 76.20 |
| 4-CHLORO-3-METHYLPHENOL | 14 | 58 | 101 | 92 | 126 | 78.2 | 43.4 | 55.46 |
| 2,4-DINITROPHENOL | 16 | 12 | 6 | 22 | 31 | 17.4 | 9.6 | 55.06 |
| 4,6-DINITRO-O-CRESOL | 12 | 6 | 61 | 252 | . | 82.8 | 115.5 | 139.57 |
| PENTACHLOROPHENOL | 13 | 60 | 31 | 66 | 209 | 75.8 | 77.5 | 102.27 |
| 4-NITROPHENOL | 42 | 113 | 115 | 86 | 18 | 74.8 | 43.3 | 57.90 |
| 2-FLUOROPHENOL | 31 | 67 | 85 | 162 | 143 | 97.6 | 54.2 | 55.51 |
| TRIFLUORO-M-CRESOL | 15 | 59 | 49 | 60 | 96 | 56.6 | 27.6 | 48.72 |
| PENTAFLUOROPHENOL | 24 | 40 | 93 | 174 | 67 | 79.6 | 59.0 | 74.08 |
| 2-FLUORONAPHTHALENE | 68 | 79 | 87 | 87 | 100 | 84.2 | 11.8 | 13.99 |
| 2,2'-DIFLUOROBIPHENYL | 72 | 92 | 95 | 73 | 98 | 86.0 | 12.5 | 14.55 |
| 2-FLUOROBIPHENYL | 76 | 92 | 83 | 62 | 84 | 79.4 | 11.3 | 14.18 |

## Key to Figure IV-1.
## GC/MS chromatogram of purgeable organics by purge and trap— Method Blank, spiked

| Symbol | Purgeables |
|---|---|
| A | Methylene chloride |
| B | Trichlorofluoromethane |
| C | 1,1-Dichloroethylene |
| D | trans-1,2-Dichloroethylene |
| E | 1,1-Dichloroethane |
| F | Chloroform |
| G | 1,2-Dichloroethane |
| H | 1,1,1-Trichloroethane |
| I | 1,1,2-Trichloroethane |
| J | Carbon tetrachloride |
| K | 1,2-Dichloropropane |
| L | trans-1,3-Dichloropropene |
| M | cis-1,3-Dichloropropene |
| N | Bromodichloromethane |
| O | Dibromochloromethane |
| P | Chlorobenzene |
| Q | Trichloroethylene |
| R | Benzene |
| S | Bromoform |
| T | 1,1,2,2-Tetrachloroethene |
| U | 1,1,2,2-Tetrachloroethane |
| V | Ethylbenzene |
| W | Toluene |

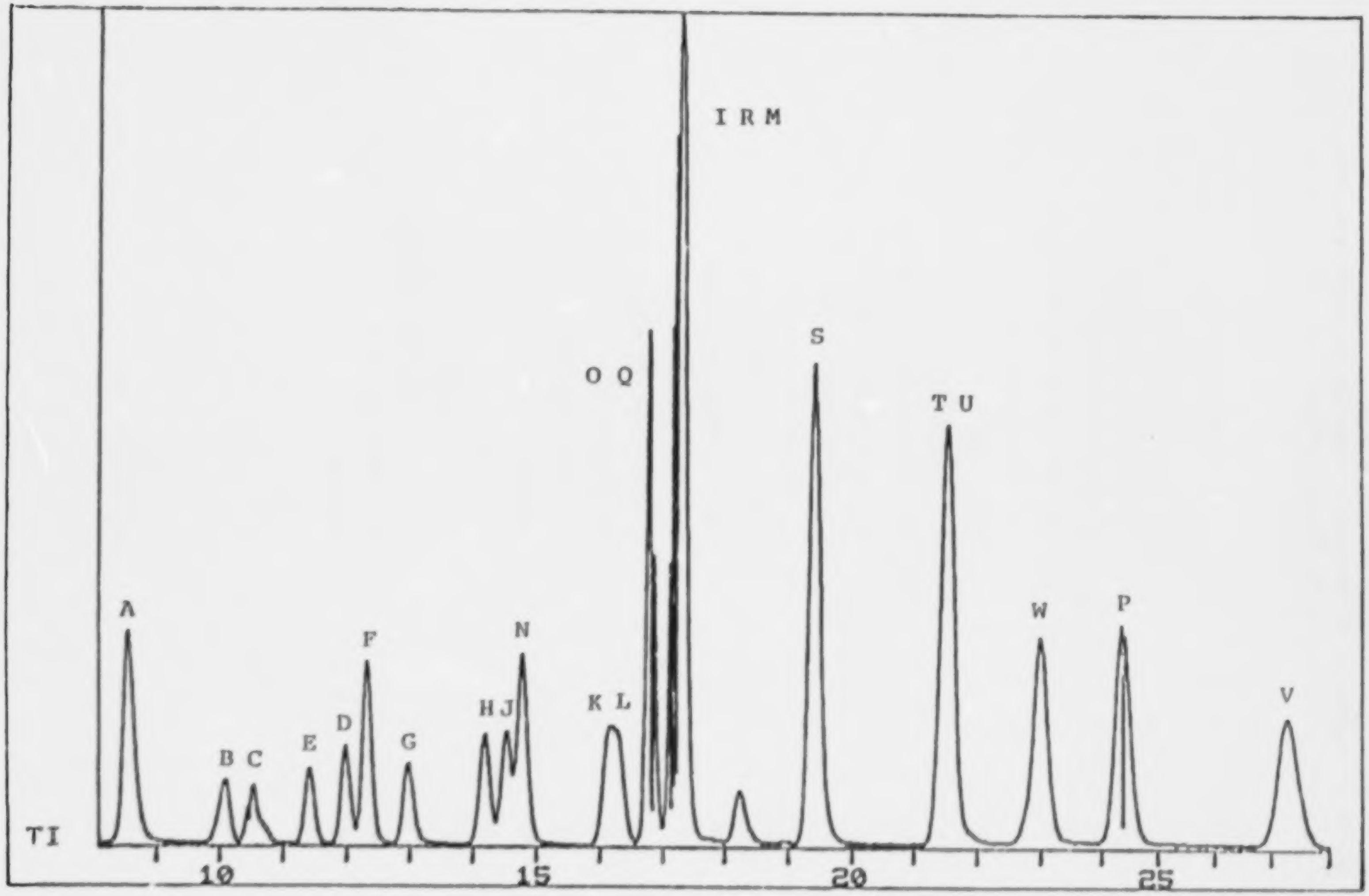


Figure IV-1. GC/MS chromatogram of purgeable organics by purge and trap—Method Blank, spiked

## Key to Figure IV-2.
## GC/MS chromatogram of base/neutral extractables — Method Blank, spiked

| Symbol | Base/neutrals |
|---|---|
| A | 1,3-Dichlorobenzene |
| B | 1,4-Dichlorobenzene |
| C | 1,2-Dichlorobenzene |
| D | Hexachloroethane |
| E | Bis(2-chloroethyl) ether |
| F | Bis(2-chloroisopropyl) ether |
| G | Hexachlorobutadiene |
| H | Nitrobenzene |
| I | Naphthalene |
| J | 1,2,4-Trichlorobenzene |
| K | Bis(2-chloroethoxy) methane |
| L | N-nitrosodipropylamine |
| M | Hexachlorocyclopentadiene |
| N | 2-Chloronaphthalene |
| O | Isophorone |
| P | Acenaphthylene |
| Q | Acenaphthene |
| R | Dimethyl phthalate |
| S | 2,6-Dinitrotoluene |
| T | Fluorene |
| U | 2,4-Dinitrotoluene |
| V | 1,2-Diphenylhydrazine |
| W | Diethyl phthalate |
| X | N-Nitrosodiphenylamine |
| Y | Hexachlorobenzene |
| Z | 4-Bromophenyl phenyl ether |
| AA/BB | Phenanthrene/anthracene |
| CC | Di-n-butyl phthalate |
| DD | Fluoranthrene |
| EE | Pyrene |
| FF | Benzidine |
| GG | Butylbenzyl phthalate |
| HH | Bis(2-ethylhexyl) phthalate |
| II/JJ | Chrysene/benzo(a)anthracene |
| KK | 3,3-Dichlorobenzidine |
| LL | Di-n-octyl phthalate |
| MM/NN | Benzo(b)fluoranthene/benzo(k)fluoranthene |
| OO | Benzo(a)pyrene |
| PP | Indeno(1,2,3-cd) pyrene |
| QQ | Dibenzo(ah)anthracene |
| RR | Benzo(ghi)perylene |

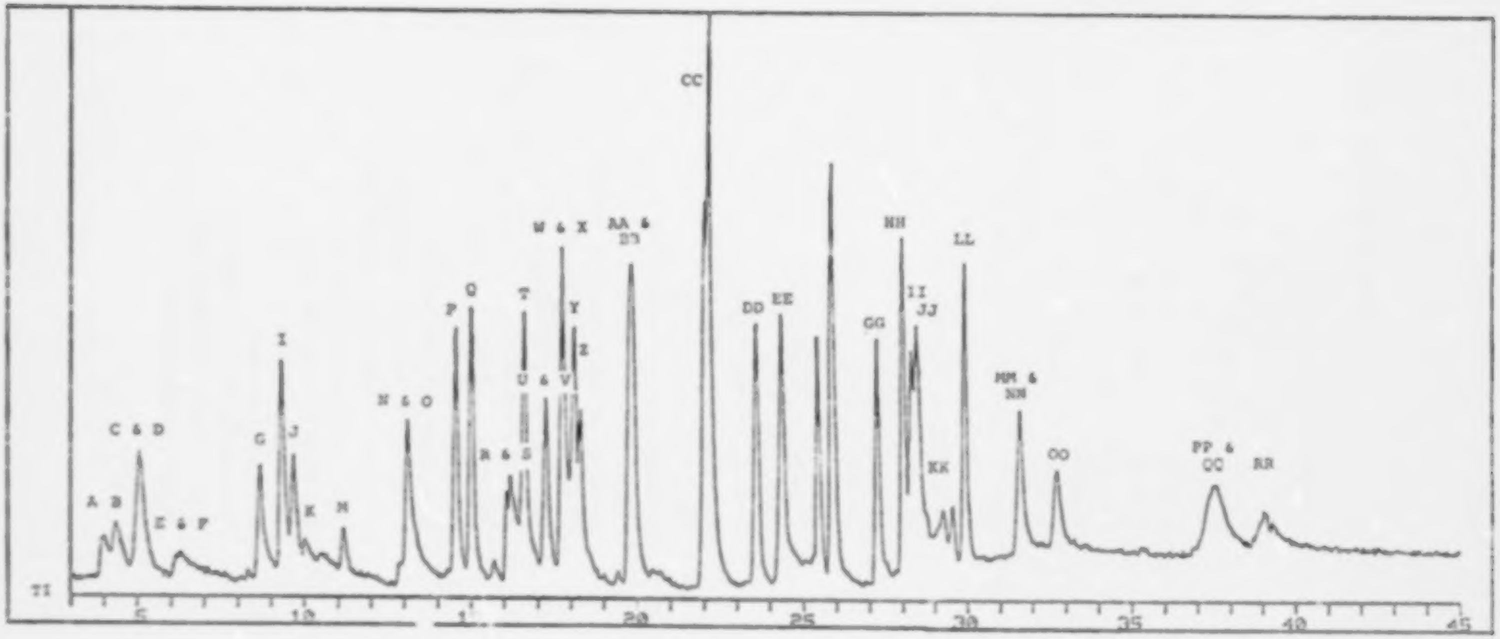


Figure IV-2. GC/MS chromatogram of base/neutral extractables—Method Blank, spiked

Key to Figure IV-3.
GC/MS chromatogram of acid extractables —
Method Blank, spiked

| Symbol | Acids |
|---|---|
| A | 2-Chlorophenol |
| B | 2-Nitrophenol |
| C | Phenol |
| D | 2,4-Dimethylphenol |
| E | 2,4-Dichlorophenol |
| F | 2,4,6-Trichlorophenol |
| G | 4-Chloro-3-methylphenol |
| H | 2,4-Dinitrophenol |
| I | Pentachlorophenol |
| J | 4-Nitrophenol |

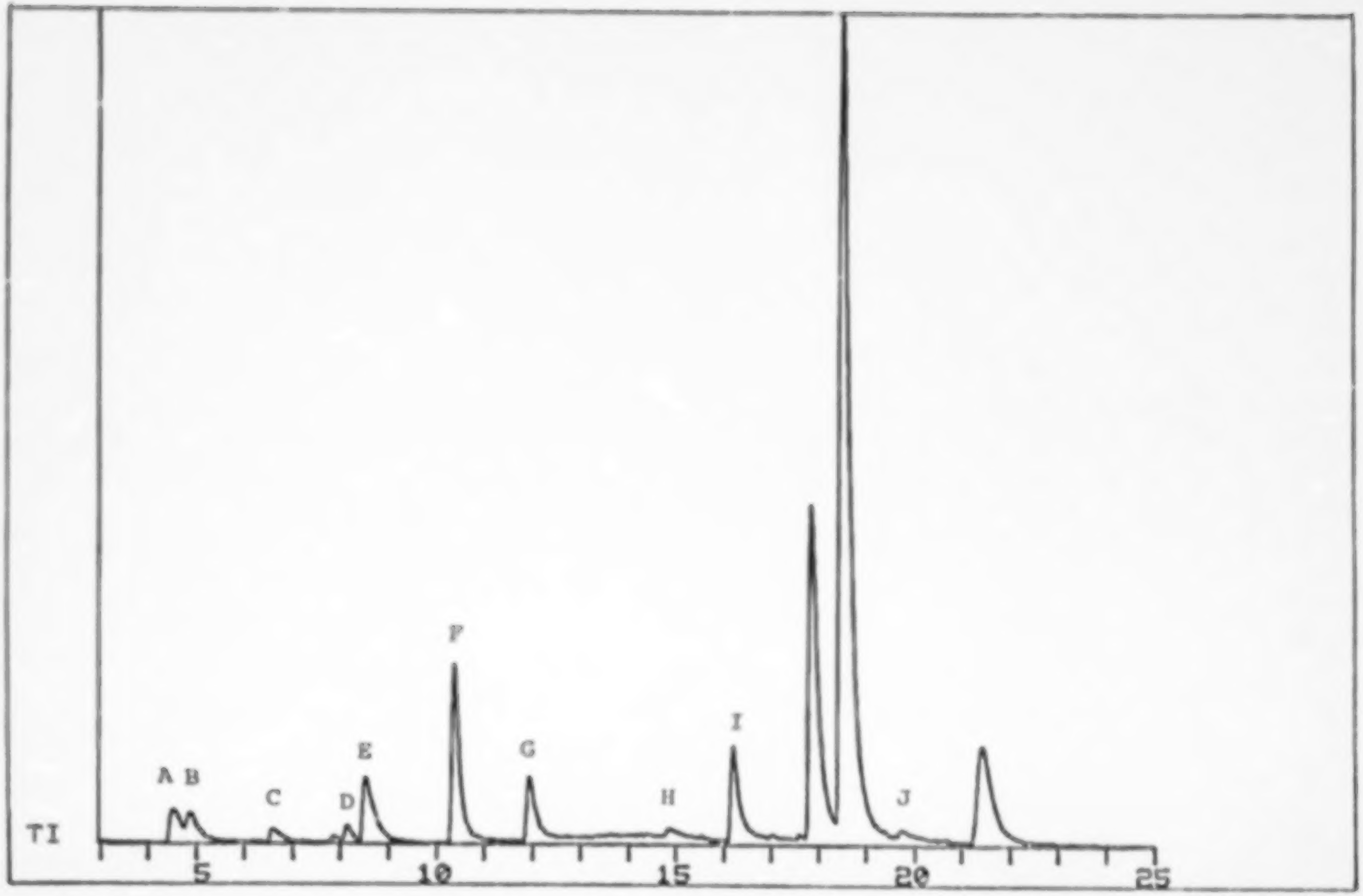


Figure IV-3. GC/MS chromatogram of acid extractables—
Method Blank, spiked

## B. POTW Residual Waste

## TABLE IV-2. PURGEABLE ORGANICS DATA-- POTW RESIDUAL WASTE, SPIKE LEVEL 0

| COMPOUND | ADDED | NO_1 | NO_2 | NO_3 | NO_4 | NO_5 | MEAN | S | RSD | RECOVERY |
|---|---|---|---|---|---|---|---|---|---|---|
| METHYLENE CHLORIDE | 0 | 0.59 | 0.1 | 1.3 | 0.78 | 0.34 | 0.662 | 0.41 | 61.4 | . |
| [illegible] | 0 | 0.04 | 0.03 | 0.02 | 0.02 | 0.03 | 0.032 | 0.01 | 24.0 | . |
| [illegible] | 0 | 0.2 | 0.2 | 0. | 0.2 | 0. | 0.22 | 0.03 | 13.6 | . |
| [illegible] | 0 | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | . |
| [illegible] | 0 | 1. | 0.0 | 0.4 | 0.41 | 0.41 | [illegible] | [illegible] | [illegible] | . |
| [illegible] | 0 | 0.04 | 1.2 | 0.4 | 0.04 | 0.08 | 1.004 | 0.14 | 13.9 | . |
| ETHYLBENZENE | 0 | 1.1 | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] |

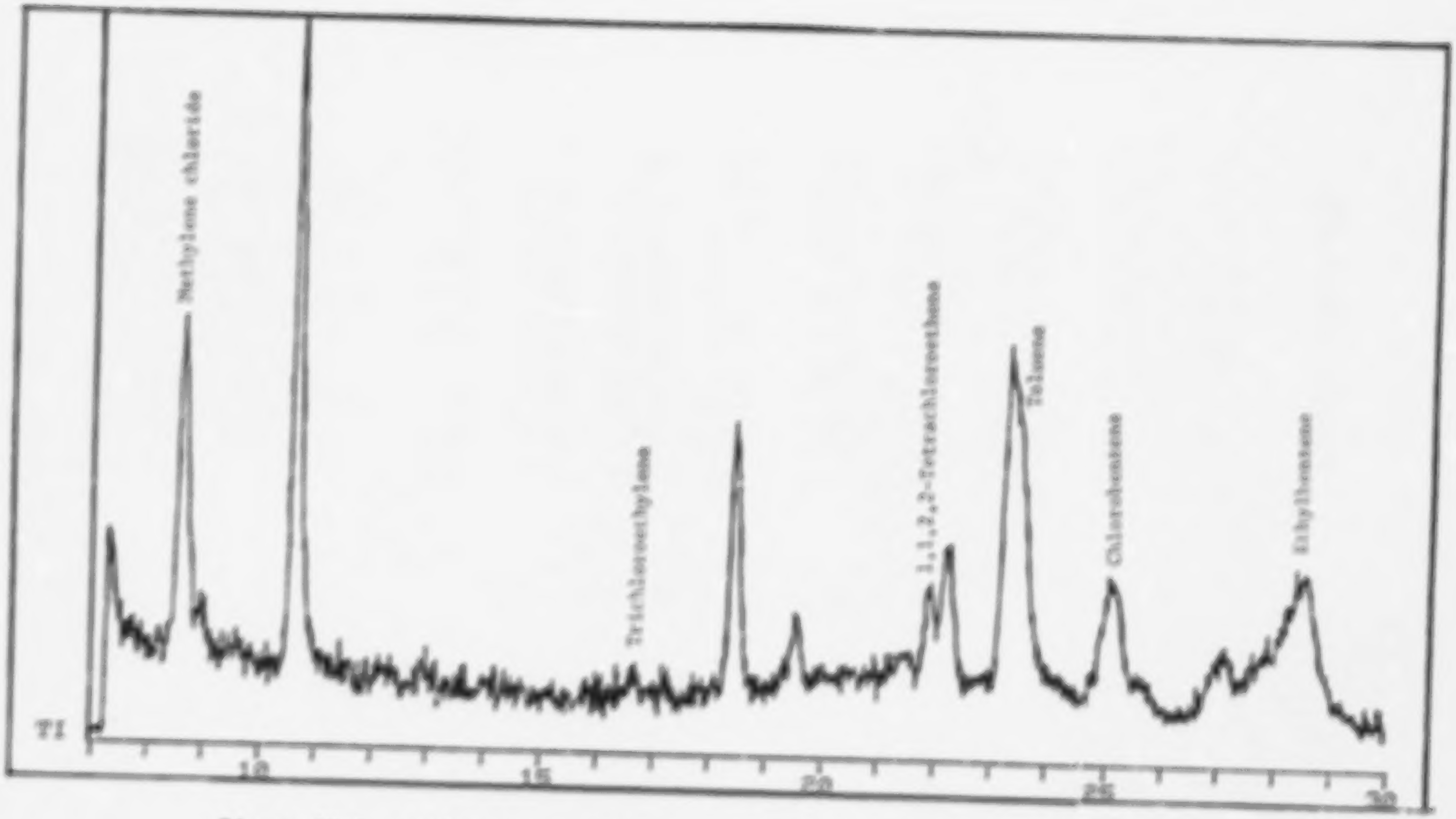


Figure IV-4. GC/MS chromatogram of purgeable organics by purge and trap—POTW Residual Waste, unspiked

# TABLE IV-3. PURGEABLE ORGANICS DATA— POTW RESIDUAL WASTE, SPIKE LEVEL I

| COMPOUND | ADDED | NO_1 | NO_2 | NO_3 | NO_4 | NO_5 | MEAN | S | RSD | RECOVERY |
|---|---|---|---|---|---|---|---|---|---|---|
| METHYLENE CHLORIDE | 0.99 | 1.7 | 0.72 | 0.65 | 0.81 | 1.1 | 0.996 | 0.43 | 43.1 | 34 |
| 1,1-DICHLOROETHYLENE | 1 | (0.42) | 0.2 | 0.18 | 0.17 | 0.18 | 0.1825 | 0.01 | 6.9 | 18 |
| 1,1-DICHLOROETHANE | 1 | (0.64) | 0.37 | 0.32 | 0.32 | 0.3 | 0.3275 | 0.03 | 9.1 | 33 |
| TRANS-1,2-DICHLOROETHYLENE | 1 | 0.5 | 0.32 | 0.28 | 0.24 | 0.29 | 0.326 | 0.10 | 31.1 | 33 |
| CHLOROFORM | 1 | (0.66) | 0.42 | 0.4 | 0.38 | 0.36 | 0.39 | 0.03 | 6.6 | 39 |
| 1,2-DICHLOROETHANE | 1 | 1.1 | 0.42 | 0.7 | 0.46 | 0.52 | 0.64 | 0.28 | 43.5 | 64 |
| 1,1,1-TRICHLOROETHANE | 1 | (0.65) | 0.42 | 0.42 | 0.39 | 0.41 | 0.41 | 0.01 | 3.4 | 41 |
| CARBON TETRACHLORIDE | 1 | 0.71 | 0.52 | 0.48 | 0.35 | 0.44 | 0.5 | 0.13 | 26.6 | 50 |
| BROMODICHLOROMETHANE | 0.99 | 0.79 | 0.56 | 0.61 | 0.47 | 0.57 | 0.6 | 0.12 | 19.6 | 61 |
| 1,2-DICHLOROPROPANE | 1 | 0.44 | 0.48 | 0.46 | 0.41 | 0.4 | 0.438 | 0.03 | 7.6 | 44 |
| TRANS-1,3-DICHLOROPROPENE | 1 | 0.24 | 0.12 | 0.16 | 0.088 | 0.17 | 0.1556 | 0.06 | 36.9 | 16 |
| TRICHLOROETHYLENE | 0.99 | 0.66 | 0.53 | 0.55 | 0.53 | 0.53 | 0.56 | 0.06 | 10.1 | 53 |
| DIBROMOCHLOROMETHANE | 1 | 0.61 | 0.52 | 0.58 | 0.51 | 0.61 | 0.566 | 0.05 | 8.5 | 57 |
| CIS-1,3-DICHLOROPROPENE | 0.81 | 0.46 | 0.12 | 0.16 | 0.1 | 0.37 | 0.242 | 0.16 | 67.2 | 30 |
| 1,1,2-TRICHLOROETHANE | 1 | 0.74 | 0.6 | 0.64 | 0.58 | 0.62 | 0.636 | 0.06 | 9.8 | 64 |
| BENZENE | 1 | (0.78) | 0.53 | 0.44 | 0.43 | 0.42 | 0.455 | 0.05 | 11.1 | 45 |
| BROMOFORM | 1 | 0.49 | 0.49 | 0.53 | 0.47 | 0.56 | 0.508 | 0.04 | 7.2 | 51 |
| 1,1,2,2-TETRACHLOROETHENE | 1 | 1.1 | 1.1 | 1.2 | 1.1 | 1.2 | 1.14 | 0.05 | 4.8 | 92 |
| 1,1,2,2-TETRACHLOROETHANE | 1 | 0.94 | 0.98 | 1 | 0.94 | 1.1 | 0.992 | 0.07 | 6.6 | 99 |
| TOLUENE | 1 | 2.2 | 1.9 | 1.9 | 1.7 | 1.9 | 1.92 | 0.18 | 9.3 | 103 |
| CHLOROBENZENE | 1 | 1.2 | 1.3 | 1.4 | 1.3 | 1.4 | 1.32 | 0.08 | 6.3 | 78 |
| ETHYLBENZENE | 1 | 1.8 | 2 | 2.1 | 1.8 | 2 | 1.94 | 0.13 | 6.9 | 94 |

# TABLE IV-4. PURGEABLE ORGANICS DATA— POTW RESIDUAL WASTE, SPIKE LEVEL II

| COMPOUND | ADDED | NO_1 | NO_2 | NO_3 | NO_4 | NO_5 | MEAN | S | RSD | RECOVERY |
|---|---|---|---|---|---|---|---|---|---|---|
| METHYLENE CHLORIDE | 9.9 | 5.6 | 5.5 | (3.1) | 6.1 | 6.6 | 5.95 | 0.51 | 8.5 | 53 |
| 1,1-DICHLOROETHYLENE | 9.9 | 5.3 | 2.6 | 3.8 | 3.6 | 4.7 | 4 | 1.04 | 26.0 | 40 |
| 1,1-DICHLOROETHANE | 10 | 5.2 | 6.2 | 5.3 | 6.3 | 7.7 | 6.14 | 1.01 | 16.4 | 61 |
| TRANS-1,2-DICHLOROETHYLENE | 10 | 4.3 | 4 | 3.6 | 4.2 | 5.4 | 4.3 | 0.67 | 15.6 | 43 |
| CHLOROFORM | 9.9 | 6.2 | 5.9 | 5.1 | 6.2 | 7.7 | 6.22 | 0.94 | 15.1 | 63 |
| 1,2-DICHLOROETHANE | 10 | 5.9 | 5.7 | 4 | 3.8 | 6.6 | 5.2 | 1.23 | 23.7 | 52 |
| 1,1,1-TRICHLOROETHANE | 10 | 7.3 | 5 | 5.5 | 5.3 | 7.8 | 6.18 | 1.28 | 20.6 | 62 |
| CARBON TETRACHLORIDE | 10 | 6.4 | 4.2 | 5.1 | 4.7 | 7 | 5.48 | 1.18 | 21.5 | 55 |
| BROMODICHLOROMETHANE | 9.9 | 8.3 | 6.2 | 4.9 | 6.5 | 7.4 | 6.66 | 1.28 | 19.2 | 67 |
| 1,2-DICHLOROPROPANE | 10 | 6 | 7.4 | 6 | 5.4 | 7.5 | 6.46 | 0.94 | 14.5 | 65 |
| TRANS-1,3-DICHLOROPROPENE | 10 | 9 | 8.7 | 7.4 | 8.8 | 11 | 8.98 | 1.29 | 14.4 | 90 |
| TRICHLOROETHYLENE | 9.9 | 8.7 | 6.5 | 5.1 | 5 | 6.2 | 6.3 | 1.49 | 23.7 | 63 |
| DIBROMOCHLOROMETHANE | 10 | 7.3 | 6.8 | 5.8 | 5.9 | 7.9 | 6.7 | 0.90 | 13.4 | 67 |
| CIS-1,3-DICHLOROPROPENE | 8.1 | 5.8 | 6.1 | 5.5 | 5.6 | 7.5 | 6.1 | 0.82 | 13.4 | 75 |
| 1,1,2-TRICHLOROETHANE | 10 | 8.6 | 8.8 | 7.3 | 7.1 | 9 | 8.16 | 0.89 | 10.9 | 82 |
| BENZENE | 9.9 | 6.1 | 6.6 | 5.7 | 5.9 | 8.8 | 6.62 | 1.26 | 19.1 | 67 |
| BROMOFORM | 10 | 8.9 | 8 | 7 | 7 | 8.8 | 7.94 | 0.93 | 11.7 | 79 |
| 1,1,2,2-TETRACHLOROETHENE | 10 | 19 | 12 | 8.1 | 6.2 | 7.1 | 10.48 | 5.25 | 50.1 | 103 |
| 1,1,2,2-TETRACHLOROETHANE | 9.9 | 15 | 11 | 6.1 | 5.5 | 7.1 | 8.94 | 4.01 | 44.8 | 90 |
| TOLUENE | 10 | 8.6 | 9.1 | 8 | 9.5 | (14) | 8.8 | 0.65 | 7.4 | 79 |
| CHLOROBENZENE | 9.5 | 6.1 | 6.9 | 4.9 | 5.3 | 8.7 | 6.38 | 1.51 | 23.6 | 62 |
| ETHYLBENZENE | 10 | 7.3 | 6.3 | 6.7 | 7.1 | 8.1 | 7.1 | 0.68 | 9.6 | 61 |

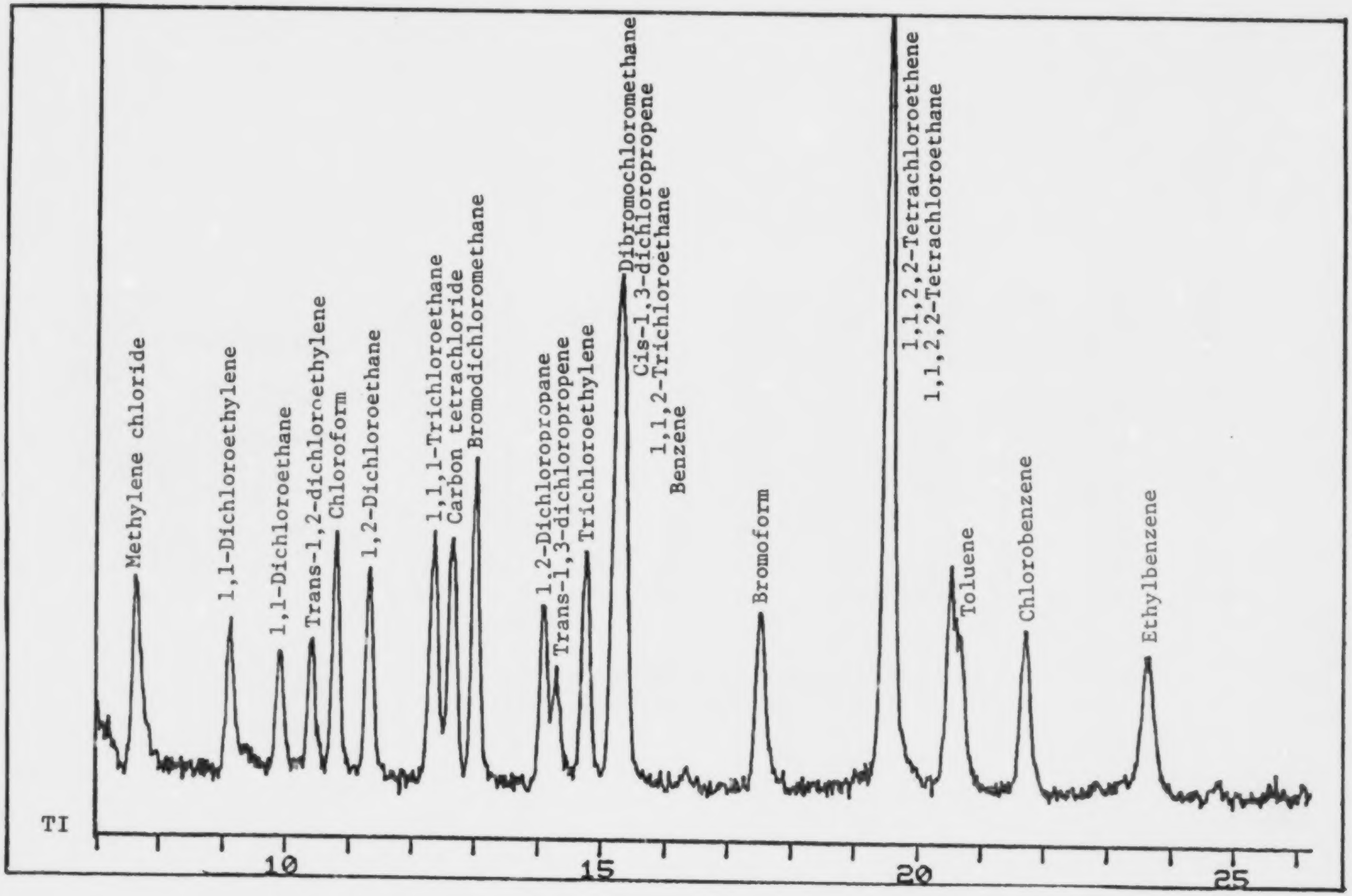


Figure IV-5. GC/MS chromatogram of purgeable organics by purge and trap—POTW Residual Waste, spiked

## TABLE IV-5. BASE/NEUTRAL EXTRACTABLE SEMIVOLATILE ORGANICS DATA— POTW RESIDUAL WASTE, SPIKE LEVEL 0

| COMPOUND | ADDED | NO_1 | NO_2 | NO_3 | NO_4 | NO_5 | MEAN | S | RSD | RECOVERY |
|---|---|---|---|---|---|---|---|---|---|---|
| 1,3-DICHLOROBENZENE | 0 | 1.6 | 2.5 | 1.8 | 2 | 2.5 | 2.08 | 0.41 | 19.6 | . |
| 1,4-DICHLOROBENZENE | 0 | 3.1 | 4.5 | 3.6 | 3.8 | 4.4 | 3.88 | 0.58 | 15.0 | . |
| 1,2-DICHLOROBENZENE | 0 | 0.59 | 0.95 | 0.75 | 0.65 | 1 | 0.788 | 0.18 | 23.0 | . |
| HEXACHLOROETHANE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| BIS(2-CHLOROETHYL) ETHER | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| BIS(2-CHLOROISOPROPYL) ETHER | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| HEXACHLOROBUTADIENE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| NITROBENZENE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| NAPHTHALENE | 0 | 4.6 | 6.4 | 5.7 | 6.8 | 7.1 | 6.12 | 1.00 | 16.3 | . |
| 1,2,4-TRICHLOROBENZENE | 0 | 40 | 61 | 52 | 57 | 59 | 53.8 | 8.41 | 15.6 | . |
| BIS(2-CHLOROETHOXY)METHANE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| N-NITROSODI-N-PROPYLAMINE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| HEXACHLOROCYCLOPENTADIENE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| 2-CHLORONAPHTHALENE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| ISOPHORONE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| ACENAPHTHYLENE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| ACENAPHTHENE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| DIMETHYL PHTHALATE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| 2,6-DINITROTOLUENE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| FLUORENE | 0 | 1.4 | 1.3 | 1.4 | 1.3 | 1.1 | 1.3 | 0.12 | 9.4 | . |
| 2,4-DINITROTOLUENE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| 1,2-DIPHENYLHYDRAZINE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| 4-CHLOROPHENYL PHENYL ETHER | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| DIETHYL PHTHALATE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| N-NITROSODIPHENYLAMINE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| HEXACHLOROBENZENE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| 4-BROMOPHENYL PHENYL ETHER | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| PHENANTHRENE/ANTHRACENE | 0 | 5.1 | 8.1 | 6.4 | 7.3 | 6.8 | 6.74 | 1.11 | 16.5 | . |
| DI-N-BUTYL PHTHALATE | 0 | 1.8 | 1.7 | 2.1 | 2.1 | 2.4 | 2.02 | 0.28 | 13.7 | . |
| FLUORANTHENE | 0 | 2.5 | 3.9 | 3.3 | 3.7 | 3.8 | 3.44 | 0.57 | 16.6 | . |
| PYRENE | 0 | 2.3 | 3.2 | 2.7 | 2.9 | 2.7 | 2.76 | 0.33 | 11.9 | . |
| BENZIDINE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| BUTYLBENZYL PHTHALATE | 0 | 7.2 | 11 | 8.6 | 12 | 8.6 | 9.48 | 1.96 | 20.7 | . |
| BIS(2-ETHYLHEXYL) PHTHALATE | 0 | 36 | 30 | 46 | 48 | 49 | 41.8 | 8.38 | 20.0 | . |
| CHRYSENE/BENZO(A)ANTHRACENE | 0 | 1.7 | 2.4 | 2.1 | 2.1 | 2 | 2.06 | 0.25 | 12.2 | . |
| 3,3'-DICHLOROBENZIDINE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| DI-N-OCTYL PHTHALATE | 0 | 2.1 | 2.7 | 2.4 | 2.6 | 2.7 | 2.5 | 0.25 | 10.2 | . |
| BENZO(B)/BENZO(K)FLUORANTHENES | 0 | 2.7 | 3.9 | 3.3 | 3.7 | 3.5 | 3.42 | 0.46 | 13.5 | . |
| BENZO(A)PYRENE | 0 | 1.4 | 1.8 | 1.4 | 1.7 | 1.5 | 1.56 | 0.18 | 11.6 | . |
| INDENO(1,2,3-CD)PYRENE | 0 | (0.49) | 0.97 | 0.82 | 0.82 | 1 | 0.9025 | 0.10 | 10.6 | . |
| DIBENZO(AH)ANTHRACENE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| BENZO(GHI)PERYLENE | 0 | <0.5 | 0.73 | 0.57 | 0.85 | 0.89 | <0.708 | 0.17 | 24.1 | . |

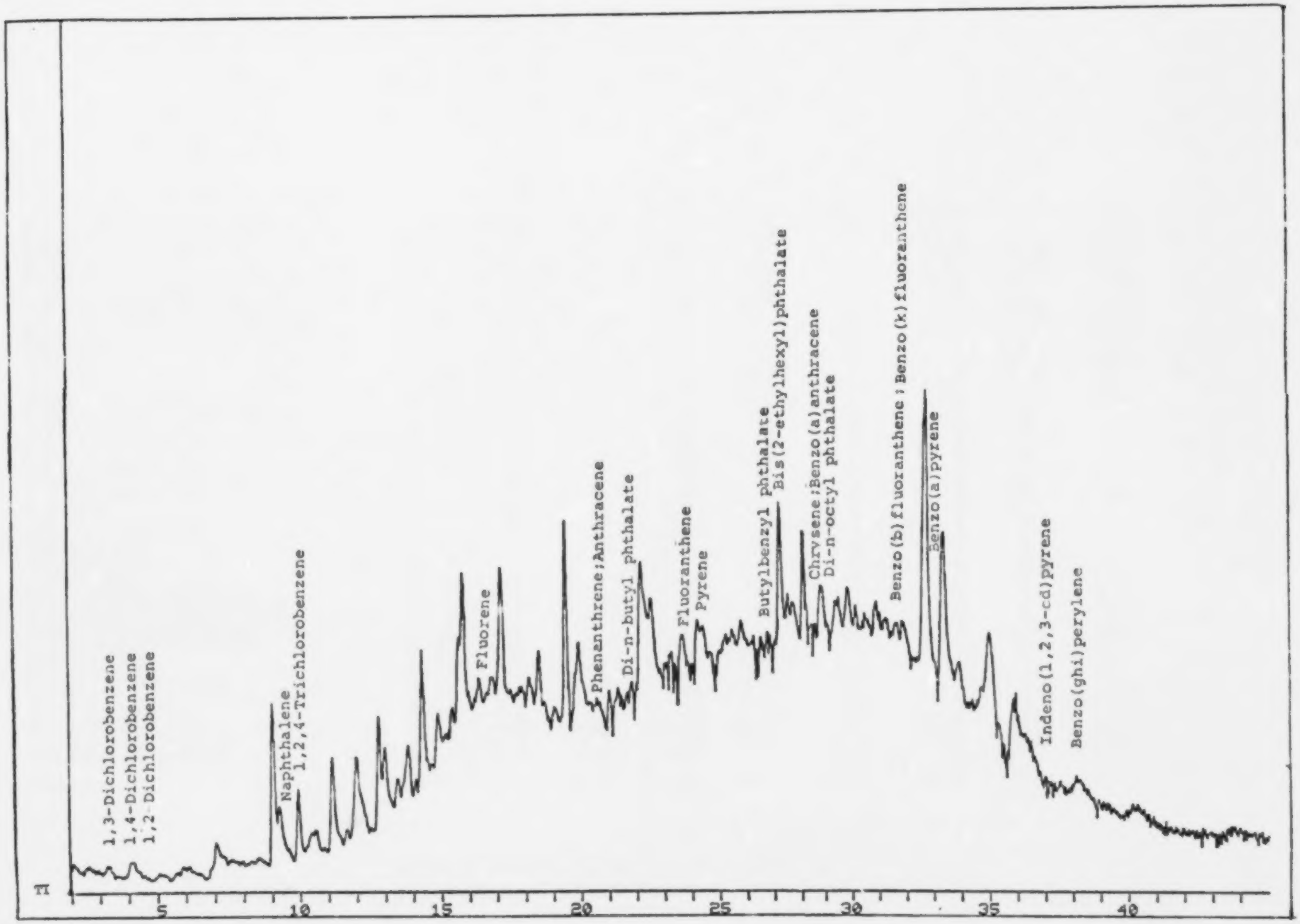


Figure IV-6. GC/MS chromatogram of base/neutral extractables — POTW Residual Waste, unspiked

TABLE IV-6. BASE/NEUTRAL EXTRACTABLE SEMIVOLATILE ORGANICS DATA—
POTW RESIDUAL WASTE, SPIKE LEVEL I

| COMPOUND | ADDED | NO_1 | NO_2 | NO_3 | NO_4 | NO_5 | MEAN | S | RSD | RECOVERY |
|---|---|---|---|---|---|---|---|---|---|---|
| 1,3-DICHLOROBENZENE | 4.3 | 13 | 13 | 8.4 | 16 | 15 | 13.08 | 2.92 | 22.3 | 256 |
| 1,4-DICHLOROBENZENE | 5.5 | 12 | 9.7 | 10 | 15 | 10 | 11.34 | 2.24 | 19.8 | 136 |
| 1,2-DICHLOROBENZENE | 5 | 5.3 | 5 | 3.8 | 5.8 | 5 | 4.98 | 0.74 | 14.8 | 84 |
| HEXACHLOROETHANE | 5 | 12 | 9.2 | 7 | 12 | 11 | 10.24 | 2.14 | 20.9 | 205 |
| BIS(2-CHLOROETHYL) ETHER | 5 | 9.5 | 7.2 | 6.6 | 4.7 | 5.8 | 6.76 | 1.80 | 26.6 | 135 |
| BIS(2-CHLOROISOPROPYL) ETHER | 5 | 5.9 | 4.5 | 2.7 | 4.2 | 5.8 | 4.02 | 2.04 | 50.7 | 80 |
| HEXACHLOROBUTADIENE | 5.1 | 3.9 | 4.7 | 3.7 | (6.1) | 4.5 | 4.2 | 0.48 | 11.3 | 82 |
| NITROBENZENE | 5 | <0.5 | <0.5 | <0.5 | <0.5 | <0.5 | <0.5 | Z | . | 10 |
| NAPHTHALENE | 5 | 12 | 9.2 | 9.2 | 12 | 15 | 11.48 | 2.41 | 21.0 | 107 |
| 1,2,4-TRICHLOROBENZENE | 39.9 | 108 | 114 | 97 | 170 | 125 | 122.8 | 28.26 | 23.0 | 173 |
| BIS(2-CHLOROETHOXY)METHANE | 5 | 2.8 | 4.6 | 2.5 | 3.4 | 5.2 | 3.7 | 1.16 | 31.4 | 74 |
| N-NITROSODI-N-PROPYLAMINE | 5 | 16 | 14 | 8.8 | 15 | 17 | 14.16 | 3.20 | 22.6 | 283 |
| HEXACHLOROCYCLOPENTADIENE | 5.1 | <0.5 | <0.5 | <0.5 | <0.5 | <0.5 | <0.5 | Z | . | 10 |
| 2-CHLORONAPHTHALENE | 4.8 | 4.8 | 5.1 | 3.7 | 7.7 | 7.9 | 5.84 | 1.86 | 31.9 | 122 |
| ISOPHORONE | 5 | 2.7 | 3.3 | 2.8 | 3 | 3.8 | 3.12 | 0.44 | 14.2 | 62 |
| ACENAPHTHYLENE | 5 | 3.8 | 5.1 | 4 | 6.3 | 6.1 | 5.06 | 1.15 | 22.8 | 101 |
| ACENAPHTHENE | 5 | (9.7) | 5.5 | 5 | 5.1 | 5.4 | 5.25 | 0.24 | 4.5 | 105 |
| DIMETHYL PHTHALATE | 5 | 4.2 | 5.8 | 3.8 | 6.3 | 8.1 | 5.64 | 1.73 | 30.7 | 113 |
| 2,6-DINITROTOLUENE | 5 | 2.2 | 2.6 | 2 | 5.3 | 3.7 | 3.16 | 1.36 | 43.2 | 63 |
| FLUORENE | 5 | 5.4 | 6.3 | 3.9 | 7.6 | 9.4 | 6.52 | 2.10 | 32.2 | 104 |
| 2,4-DINITROTOLUENE | 5.1 | 5.2 | 4.4 | 0.74 | 1.4 | 3 | 2.948 | 1.90 | 64.5 | 58 |
| 1,2-DIPHENYLHYDRAZINE | 5 | 4.9 | 4.5 | 4.2 | 5.5 | 5.4 | 4.9 | 0.56 | 11.5 | 98 |
| 4-CHLOROPHENYL PHENYL ETHER | 5 | 1.8 | 5.5 | 3 | 6.1 | 6.5 | 4.58 | 2.07 | 45.1 | 92 |
| DIETHYL PHTHALATE | 5.1 | 5.2 | 3 | 5.5 | 5.6 | 6.6 | 5.18 | 1.33 | 25.6 | 102 |
| N-NITROSODIPHENYLAMINE | 5 | 12 | 25 | 8.3 | 29 | 21 | 19.06 | 8.71 | 45.7 | 381 |
| HEXACHLOROBENZENE | 4 | 2.4 | 2.4 | 2.5 | 3.7 | 4.6 | 3.12 | 0.99 | 31.8 | 78 |
| 4-BROMOPHENYL PHENYL ETHER | 5.2 | 9.3 | 3.1 | 2.7 | 4 | 5.3 | 4.88 | 2.66 | 54.6 | 94 |
| PHENANTHRENE/ANTHRACENE | 10.1 | 14 | 15 | 15 | 16 | 17 | 15.4 | 1.14 | 7.4 | 86 |
| DI-N-BUTYL PHTHALATE | 5 | 8.3 | 7.5 | 3.6 | 6 | 8.7 | 6.82 | 2.08 | 30.4 | 96 |
| FLUORANTHENE | 5 | 8.7 | 9.4 | 7.4 | 11 | 14 | 10.1 | 2.54 | 25.1 | 133 |
| PYRENE | 5.1 | 9.9 | 9 | 7.4 | 12 | 11 | 9.86 | 1.78 | 18.1 | 139 |
| BENZIDINE | 5 | <0.5 | <0.5 | <0.5 | <0.5 | <0.5 | <0.5 | Z | . | 10 |
| BUTYL BENZYL PHTHALATE | 10.1 | 41 | 38 | 35 | 53 | 42 | 41.8 | 6.83 | 16.3 | 320 |
| BIS(2-ETHYLHEXYL) PHTHALATE | 40.2 | 156 | 128 | 128 | 149 | 164 | 145 | 16.40 | 11.3 | 257 |
| CHRYSENE/BENZO(A)ANTHRACENE | 9.7 | 57 | 47 | 56 | 54 | 72 | 57.2 | 9.15 | 16.0 | 568 |
| 3,3'-DICHLOROBENZIDINE | 5 | 6.5 | 8.9 | 6.2 | 9.2 | 8.5 | 7.86 | 1.40 | 17.9 | 157 |
| DI-N-OCTYL PHTHALATE | 5.1 | 8.4 | 6.9 | 7.3 | 8.6 | 7.6 | 7.76 | 0.72 | 9.3 | 103 |
| BENZO(B)/BENZO(K)FLUORANTHENES | 10.3 | 31 | 27 | 23 | 38 | 38 | 31.4 | 6.66 | 21.2 | 272 |
| BENZO(A)PYRENE | 5.2 | 11 | 13 | 8.9 | 15 | 12 | 11.98 | 2.27 | 18.9 | 200 |
| INDENO(1,2,3-CD)PYRENE | 5 | 18 | 19 | 15 | 22 | 23 | 19.4 | 3.21 | 16.5 | 370 |
| DIBENZO(AH)ANTHRACENE | 5 | 14 | 15 | 12 | 17 | 22 | 16 | 3.81 | 23.8 | 320 |
| BENZO(GHI)PERYLENE | 5 | 16 | 21 | 12 | 20 | 20 | 17.8 | 3.77 | 21.2 | 342 |

## TABLE IV-7. BASE/NEUTRAL EXTRACTABLE SEMIVOLATILE ORGANICS DATA — POTW RESIDUAL WASTE, SPIKE LEVEL II

| COMPOUND | ADDED | NO_1 | NO_2 | NO_3 | NO_4 | NO_5 | MEAN | S | RSD | RECOVERY |
|---|---|---|---|---|---|---|---|---|---|---|
| 1,3-DICHLOROBENZENE | 8.5 | 5.8 | 6 | 5.4 | 7.3 | 3.4 | 5.58 | 1.41 | 25.3 | 41 |
| 1,4-DICHLOROBENZENE | 11 | 4.8 | 6.3 | 3.4 | 9.5 | 5.8 | 5.96 | 2.27 | 38.1 | 19 |
| 1,2-DICHLOROBENZENE | 10 | 7.2 | 8.6 | 6.1 | 7.8 | 5.4 | 7.02 | 1.29 | 18.3 | 62 |
| HEXACHLOROETHANE | 10 | 9.8 | 12 | 6.7 | 9.2 | 5.6 | 8.66 | 2.55 | 29.4 | 87 |
| BIS(2-CHLOROETHYL) ETHER | 10 | 7.8 | 12 | <1 | <1 | <1 | <4.56 | 5.10 | 111.8 | 46 |
| BIS(2-CHLOROISOPROPYL) ETHER | 10 | 7.1 | 7.8 | 3.6 | 7.2 | 4.3 | 6 | 1.91 | 31.8 | 60 |
| HEXACHLOROBUTADIENE | 10.2 | 9.2 | 11 | 7.8 | 11 | 7.2 | 9.24 | 1.76 | 19.1 | 91 |
| NITROBENZENE | 10 | <1 | 1.6 | 1.3 | <1 | <1 | <1.18 | 0.27 | 22.7 | 12 |
| NAPHTHALENE | 10 | 10 | 14 | 9 | 11 | 6.3 | 10.06 | 2.81 | 28.0 | 39 |
| 1,2,4-TRICHLOROBENZENE | 79.7 | 145 | 179 | 134 | 160 | 109 | 145.4 | 26.44 | 18.2 | 115 |
| BIS(2-CHLOROETHOXY)METHANE | 10.1 | 3.6 | 6.3 | 3.4 | 5.2 | 2.1 | 4.12 | 1.64 | 39.9 | 41 |
| N-NITROSODI-N-PROPYLAMINE | 10 | 13 | 14 | 8.3 | 13 | 9.2 | 11.5 | 2.56 | 22.3 | 115 |
| HEXACHLOROCYCLOPENTADIENE | 10.1 | <1 | <1 | <1 | <1 | <1 | <1 | 2 | . | 10 |
| 2-CHLORONAPHTHALENE | 9.6 | 7.1 | 7.7 | 6.7 | 5.9 | 4.8 | 6.44 | 1.13 | 17.5 | 67 |
| ISOPHORONE | 10.1 | 2.2 | 2.7 | 1.7 | 2.3 | 2 | 2.18 | 0.37 | 17.0 | 22 |
| ACENAPHTHYLENE | 10.1 | 6.6 | 9 | 6.6 | 7.9 | 4.6 | 6.94 | 1.65 | 23.8 | 69 |
| ACENAPHTHENE | 10.1 | 6.7 | 8.4 | 4.2 | 6.5 | 4.3 | 6.02 | 1.78 | 29.5 | 60 |
| DIMETHYL PHTHALATE | 10.1 | 6 | 7.3 | 5.9 | 8.5 | 5.1 | 6.56 | 1.34 | 20.4 | 65 |
| 2,6-DINITROTOLUENE | 10.1 | 1.8 | 2 | 1.5 | 2.4 | 1.1 | 1.76 | 0.49 | 28.0 | 17 |
| FLUORENE | 10 | 7 | 6.8 | 5.8 | 8.1 | 6.2 | 6.78 | 0.88 | 13.0 | 55 |
| 2,4-DINITROTOLUENE | 10.2 | 1.2 | 1.6 | 1.4 | (3.9) | 0.97 | 1.2925 | 0.27 | 20.9 | 13 |
| 1,2-DIPHENYLHYDRAZINE | 10 | 4.1 | 5.4 | 2.7 | 4.6 | 2.1 | 3.78 | 1.36 | 36.0 | 38 |
| 4-CHLOROPHENYL PHENYL ETHER | 10 | 7.5 | 6.9 | 7.5 | 7.3 | 6.1 | 7.06 | 0.59 | 8.4 | 71 |
| DIETHYL PHTHALATE | 10.1 | 6.4 | 8.1 | 6.5 | 7.9 | 6.6 | 7.1 | 0.83 | 11.7 | 70 |
| N-NITROSODIPHENYLAMINE | 10 | 35 | 41 | 26 | 29 | 24 | 31 | 6.96 | 22.5 | 310 |
| HEXACHLOROBENZENE | 8.1 | 4.9 | 6.9 | 5.9 | 5.8 | 4.9 | 5.68 | 0.83 | 14.6 | 70 |
| 4-BROMOPHENYL PHENYL ETHER | 10.3 | 8.4 | 10 | 9.1 | 8.8 | 7.2 | 8.7 | 1.02 | 11.8 | 84 |
| PHENANTHRENE/ANTHRACENE | 20.1 | 17 | 21 | 14 | 15 | 12 | 15.8 | 3.42 | 21.6 | 45 |
| DI-N-BUTYL PHTHALATE | 10.1 | 8.2 | 11 | 7.2 | 8.7 | 6.2 | 8.26 | 1.81 | 21.9 | 62 |
| FLUORANTHENE | 10 | 12 | 16 | 11 | 12 | 9.8 | 12.16 | 2.33 | 19.2 | 87 |
| PYRENE | 10.1 | 13 | 17 | 12 | 14 | 8.5 | 12.9 | 3.09 | 24.0 | 100 |
| BENZIDINE | 10 | 2.4 | 2.1 | 3 | 2.2 | 2 | 2.34 | 0.40 | 17.0 | 23 |
| BUTYLBENZYL PHTHALATE | 20.2 | 41 | (82) | 38 | 35 | 24 | 34.5 | 7.42 | 21.5 | 124 |
| BIS(2-ETHYLHEXYL) PHTHALATE | 80.5 | 207 | 258 | 198 | 168 | 121 | 190.4 | 50.55 | 26.5 | 185 |
| CHRYSENE/BENZO(A)ANTHRACENE | 19.3 | 81 | 115 | 90 | 89 | 47 | 84.4 | 24.49 | 29.0 | 427 |
| 3,3'-DICHLOROBENZIDINE | 10 | 16 | 24 | 15 | 14 | 11 | 16 | 4.85 | 30.3 | 160 |
| DI-N-OCTYL PHTHALATE | 10.1 | 7.2 | 10 | 6.8 | 6 | 3.2 | 6.64 | 2.44 | 36.8 | 41 |
| BENZO(B)/BENZO(K)FLUORANTHENES | 20.6 | 37 | 55 | 43 | 40 | 25 | 40 | 10.82 | 27.0 | 178 |
| BENZO(A)PYRENE | 10.1 | 9.2 | 21 | 17 | 16 | 8.9 | 14.42 | 5.25 | 36.4 | 127 |
| INDENO(1,2,3-CD)PYRENE | 10.1 | 20 | 28 | 23 | 23 | 12 | 21.2 | 5.89 | 27.8 | 201 |
| DIBENZO(AH)ANTHRACENE | 10.1 | 20 | 27 | 20 | 19 | 12 | 19.6 | 5.32 | 27.1 | 194 |
| BENZO(GHI)PERYLENE | 10.1 | 16 | 26 | 19 | 20 | 11 | 18.4 | 5.50 | 29.9 | 175 |

## TABLE IV-8. BASE/NEUTRAL EXTRACTABLE SEMIVOLATILE ORGANICS DATA— POTW RESIDUAL WASTE, SPIKE LEVEL III

| COMPOUND | ADDED | NO_1 | NO_2 | NO_3 | NO_4 | NO_5 | MEAN | S | RSD | RECOVERY |
|---|---|---|---|---|---|---|---|---|---|---|
| 1,3-DICHLOROBENZENE | 17.1 | 18 | 18 | 12 | 14 | 19 | 16.2 | 3.03 | 10.7 | 83 |
| 1,4-DICHLOROBENZENE | 21.9 | 20 | 19 | 15 | 14 | 20 | 17.6 | 2.88 | 16.4 | 63 |
| 1,2-DICHLOROBENZENE | 20.1 | 20 | 16 | 14 | 14 | 18 | 16.4 | 2.61 | 15.9 | 78 |
| HEXACHLOROETHANE | 20 | 21 | 16 | 14 | 13 | 17 | 16.2 | 3.11 | 19.2 | 81 |
| BIS(2-CHLOROETHYL) ETHER | 20 | 31 | 26 | 18 | 18 | 21 | 22.8 | 5.63 | 24.7 | 114 |
| BIS(2-CHLOROISOPROPYL) ETHER | 20.1 | 13 | 11 | 7.4 | 6.8 | 9.8 | 9.6 | 2.56 | 26.7 | 48 |
| HEXACHLOROBUTADIENE | 20.5 | 20 | 20 | 23 | 23 | 27 | 22.6 | 2.88 | 12.7 | 110 |
| NITROBENZENE | 20.1 | 11 | 13 | 6.6 | 8.8 | 11 | 10.08 | 2.45 | 24.3 | 50 |
| NAPHTHALENE | 20 | 24 | 20 | 19 | 20 | 24 | 21.4 | 2.41 | 11.3 | 76 |
| 1,2,4-TRICHLOROBENZENE | 159.5 | 238 | 256 | 284 | 268 | 346 | 278.4 | 41.36 | 14.9 | 141 |
| BIS(2-CHLOROETHOXY)METHANE | 20.2 | 10 | 10 | 7.4 | 6.5 | 10 | 8.78 | 1.70 | 19.4 | 43 |
| N-NITROSODI-N-PROPYLAMINE | 20 | 17 | 16 | 5.5 | 21 | 8.5 | 13.6 | 6.40 | 47.0 | 68 |
| HEXACHLOROCYCLOPENTADIENE | 20.2 | <1 | <1 | <1 | <1 | <1 | <1 | 2 | . | 5 |
| 2-CHLORONAPHTHALENE | 19.2 | 20 | 17 | 16 | 15 | 20 | 17.6 | 2.30 | 13.1 | 92 |
| ISOPHORONE | 20.2 | 3.2 | 2.6 | 1.9 | 1.6 | 1.9 | 2.24 | 0.65 | 29.0 | 11 |
| ACENAPHTHYLENE | 20.2 | 17 | 16 | 15 | 15 | 19 | 16.4 | 1.67 | 10.2 | 81 |
| ACENAPHTHENE | 20.1 | 17 | 14 | 14 | 13 | 18 | 15.2 | 2.17 | 14.3 | 76 |
| DIMETHYL PHTHALATE | 20.1 | 13 | 10 | 10 | 9.8 | 13 | 11.16 | 1.68 | 15.1 | 56 |
| 2,6-DINITROTOLUENE | 20.1 | 9.6 | 7.7 | 8.3 | 8 | 9.9 | 8.7 | 0.99 | 11.3 | 43 |
| FLUORENE | 20.1 | 20 | 13 | 14 | 13 | 20 | 16 | 3.67 | 23.0 | 73 |
| 2,4-DINITROTOLUENE | 20.3 | 7.5 | 4.5 | 5 | 3.5 | 7.8 | 5.66 | 1.90 | 33.5 | 28 |
| 1,2-DIPHENYLHYDRAZINE | 20 | 7.6 | 9 | 7.4 | 6.4 | 7.2 | 7.52 | 0.94 | 12.6 | 38 |
| 4-CHLOROPHENYL PHENYL ETHER | 20.1 | 24 | 16 | 18 | 16 | 24 | 19.6 | 4.10 | 20.9 | 98 |
| DIETHYL PHTHALATE | 20.2 | 16 | 8.7 | 17 | 11 | 20 | 14.54 | 4.60 | 31.6 | 72 |
| N-NITROSODIPHENYLAMINE | 20 | (79) | 29 | 31 | 24 | 43 | 31.75 | 8.06 | 25.4 | 159 |
| HEXACHLOROBENZENE | 16 | 18 | 13 | 15 | 13 | 21 | 16 | 3.46 | 21.7 | 100 |
| 4-BROMOPHENYL PHENYL ETHER | 20.7 | 25 | 17 | 22 | 19 | 26 | 21.8 | 3.83 | 17.6 | 105 |
| PHENANTHRENE/ANTHRACENE | 40.4 | 47 | 40 | 41 | 38 | 42 | 41.6 | 3.36 | 8.1 | 86 |
| DI-N-BUTYL PHTHALATE | 20.1 | 25 | 17 | 20 | 18 | 23 | 20.6 | 3.36 | 16.3 | 92 |
| FLUORANTHENE | 20.1 | 31 | 22 | 25 | 21 | 21 | 24 | 4.24 | 17.7 | 102 |
| PYRENE | 20.2 | 30 | 18 | 23 | 21 | 30 | 24.4 | 5.41 | 22.2 | 107 |
| BENZIDINE | 20 | 20 | 11 | 9.9 | 14 | 12 | 13.38 | 4.00 | 29.9 | 67 |
| BUTYLBENZYL PHTHALATE | 40.3 | 75 | 59 | 62 | 53 | 94 | 68.6 | 16.32 | 23.8 | 147 |
| BIS(2-ETHYLHEXYL) PHTHALATE | 161 | 342 | 277 | 282 | 209 | 375 | 297 | 64.18 | 21.6 | 159 |
| CHRYSENE/BENZO(A)ANTHRACENE | 38.6 | 179 | 161 | 126 | 122 | 209 | 159.4 | 36.61 | 23.0 | 408 |
| 3,3'-DICHLOROBENZIDINE | 20 | 48 | 35 | 30 | 41 | 44 | 39.6 | 7.16 | 18.1 | 198 |
| DI-N-OCTYL PHTHALATE | 20.3 | 5.4 | 12 | 1 | 7.7 | 5.7 | 6.36 | 3.99 | 62.7 | 19 |
| BENZO(B)/BENZO(K)FLUORANTHENES | 41.1 | 87 | 72 | 62 | 70 | 78 | 73.8 | 9.34 | 12.7 | 171 |
| BENZO(A)PYRENE | 20.2 | 32 | 33 | 18 | 31 | 35 | 29.8 | 6.76 | 22.7 | 140 |
| INDENO(1,2,3-CD)PYRENE | 20.2 | 40 | 31 | 29 | 30 | 49 | 35.8 | 8.58 | 24.0 | 173 |
| DIBENZO(AH)ANTHRACENE | 20.2 | 37 | 32 | 27 | 29 | 48 | 34.6 | 8.38 | 24.2 | 171 |
| BENZO(GHI)PERYLENE | 20.1 | 34 | 31 | 28 | 29 | (50) | 30.5 | 2.65 | 8.7 | 148 |

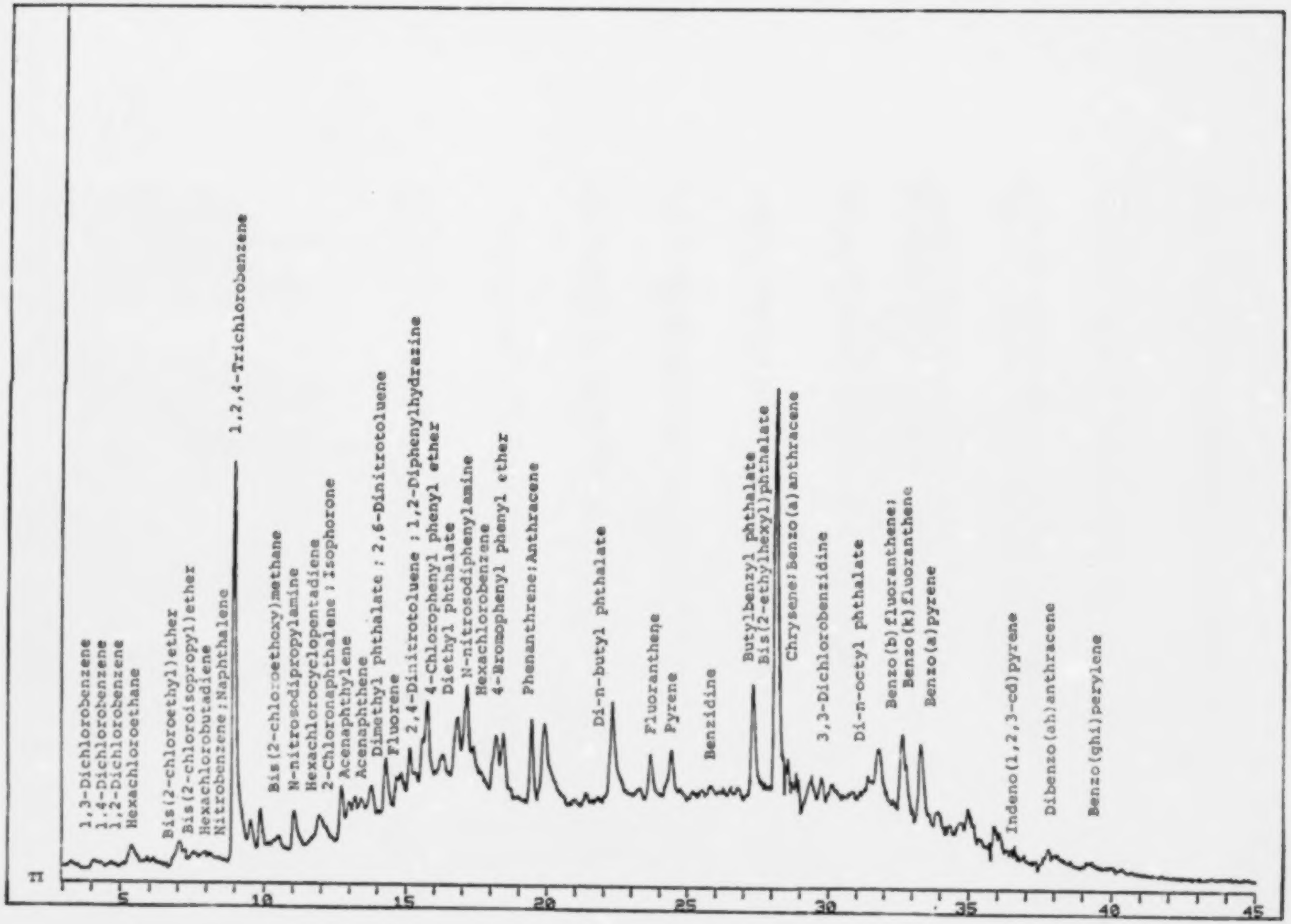


Figure IV-7. GC/MS chromatogram of base/neutral extractables—POTW Residual Waste, spiked

## TABLE IV-9. ACID EXTRACTABLE SEMIVOLATILE ORGANICS DATA— POTW RESIDUAL WASTE, SPIKE LEVEL 0

| COMPOUND | ADDED | NO_1 | NO_2 | NO_3 | NO_4 | NO_5 | MEAN | S | RSD | RECOVERY |
|---|---|---|---|---|---|---|---|---|---|---|
| 2-CHLOROPHENOL | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| 2-NITROPHENOL | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| PHENOL | 0 | 0.66 | 0.92 | 0.83 | 0.77 | 0.93 | 0.822 | 0.11 | 13.6 | . |
| 2,4-DIMETHYLPHENOL | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| 2,4-DICHLOROPHENOL | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| 2,4,6-TRICHLOROPHENOL | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| 4-CHLORO-3-METHYLPHENOL | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| 2,4-DINITROPHENOL | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| 4,6-DINITRO-O-CRESOL | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| PENTACHLOROPHENOL | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| 4-NITROPHENOL | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |

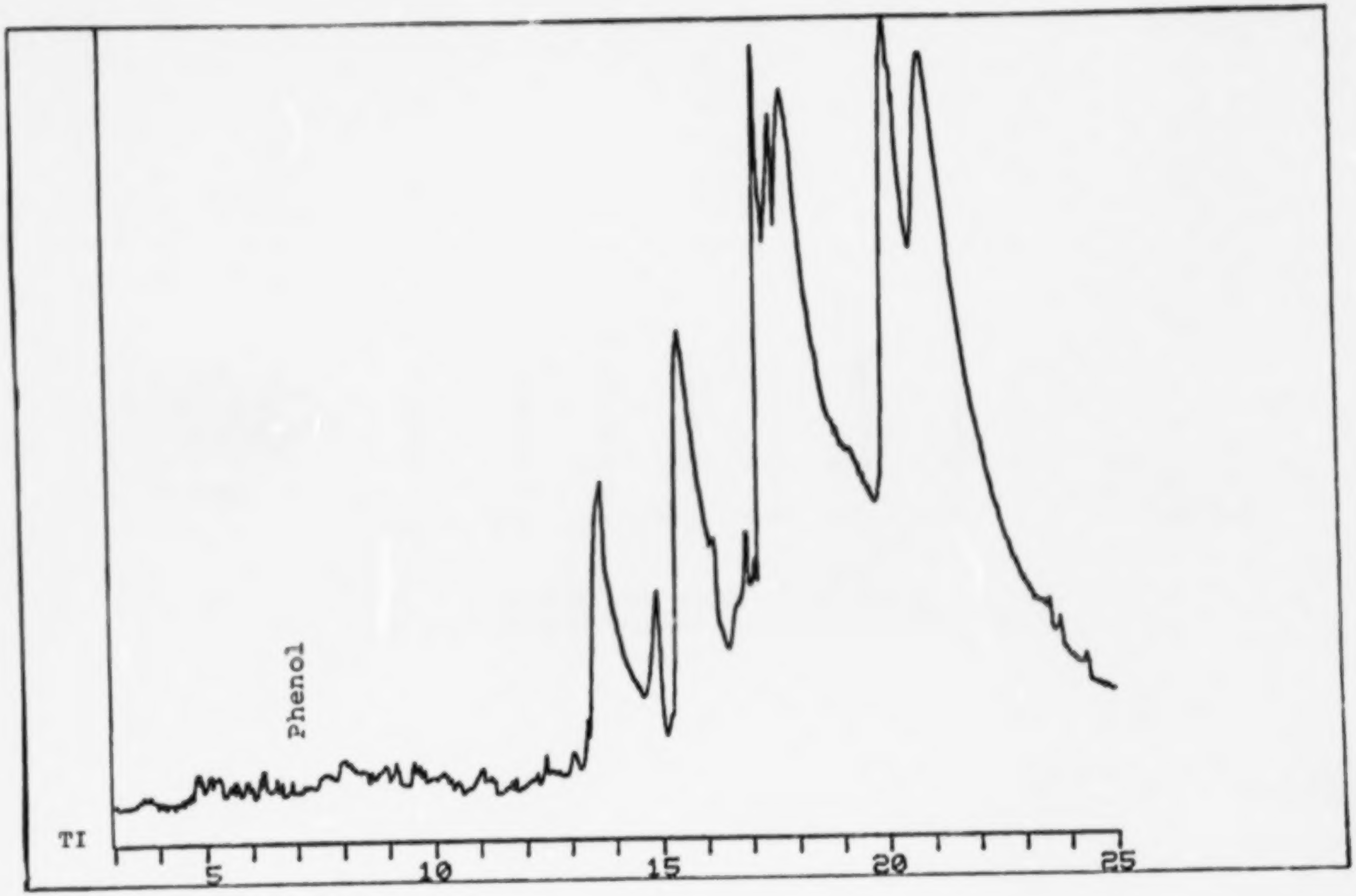


Figure IV-8. GC/MS chromatogram of acid extractables—
POTW Residual Waste, unspiked

## TABLE IV-10. ACID EXTRACTABLE SEMIVOLATILE ORGANICS DATA— POTW RESIDUAL WASTE, SPIKE LEVEL I

| COMPOUND | ADDED | NO_1 | NO_2 | NO_3 | NO_4 | NO_5 | MEAN | S | RSD | RECOVERY |
|---|---|---|---|---|---|---|---|---|---|---|
| 2-CHLOROPHENOL | 5 | 3.3 | 3.6 | 2.8 | 5.9 | 5.4 | 4.2 | 1.37 | 32.5 | 84 |
| 2-NITROPHENOL | 5 | 0.61 | 1 | 1.2 | 1.1 | (1.1) | 0.9775 | 0.26 | 26.5 | 20 |
| PHENOL | 5 | 4.8 | 5 | 3.1 | 6.6 | 5.6 | 5.02 | 1.28 | 25.5 | 84 |
| 2,4-DIMETHYLPHENOL | 5 | 1.2 | 1.9 | 1.3 | 1.6 | 2.7 | 1.74 | 0.60 | 34.6 | 35 |
| 2,4-DICHLOROPHENOL | 5.1 | 2.5 | 3 | 2.5 | 5.3 | 4.6 | 3.58 | 1.29 | 36.1 | 70 |
| 2,4,6-TRICHLOROPHENOL | 5 | 4.1 | 6.1 | 3.8 | 5 | 4.8 | 4.76 | 0.90 | 18.8 | 95 |
| 4-CHLORO-3-METHYLPHENOL | 5 | 2.5 | 1.9 | 1.7 | 3.2 | 3.2 | 2.5 | 0.70 | 28.1 | 50 |
| 2,4-DINITROPHENOL | 5 | <0.5 | <0.5 | 1.1 | 1.6 | 1.5 | <1.04 | 0.53 | 50.7 | 21 |
| 4,6-DINITRO-O-CRESOL | 5 | 4.2 | 7 | 1.9 | 3.7 | 4.8 | 4.32 | 1.85 | 42.8 | 86 |
| PENTACHLOROPHENOL | 5 | 5.9 | 8.2 | 6 | 8 | 11 | 7.82 | 2.08 | 26.6 | 156 |
| 4-NITROPHENOL | 5 | 23 | 44 | 31 | 46 | 55 | 39.8 | 12.72 | 32.0 | 796 |

## TABLE IV-11. ACID EXTRACTABLE SEMIVOLATILE ORGANICS DATA— POTW RESIDUAL WASTE, SPIKE LEVEL II

| COMPOUND | ADDED | NO_1 | NO_2 | NO_3 | NO_4 | NO_5 | MEAN | S | RSD | RECOVERY |
|---|---|---|---|---|---|---|---|---|---|---|
| 2-CHLOROPHENOL | 10 | 6.2 | 7.7 | 5.6 | 5.8 | 5.8 | 6.22 | 0.86 | 13.8 | 62 |
| 2-NITROPHENOL | 10 | 1.8 | 3.6 | 3.4 | 1.9 | 3.0 | 2.74 | 0.82 | 29.8 | 27 |
| PHENOL | 10 | 6.1 | 7.6 | 6.3 | 6.5 | 3.1 | 5.92 | 1.14 | 19.2 | 59 |
| 2,4-DIMETHYLPHENOL | 10 | 3.9 | 2.6 | 3.8 | 2.1 | 4.5 | 3.4 | 0.96 | 28.1 | 34 |
| 2,4-DICHLOROPHENOL | 10 | 6.3 | 7.6 | 5.6 | 5.1 | 4.2 | 5.76 | 1.28 | 22.3 | 57 |
| 2,4,6-TRICHLOROPHENOL | 10 | 8.2 | 7.9 | 5.8 | 10 | 4.5 | 7.28 | 2.15 | 29.6 | 73 |
| 4-CHLORO-3-METHYLPHENOL | 10 | 3.8 | 4.4 | 3 | 3.3 | 5 | 3.94 | 0.78 | 19.8 | 39 |
| 2,4-DINITROPHENOL | 10 | <1 | 6.7 | 13 | 5.7 | <1 | <5.48 | 4.96 | 90.4 | 55 |
| 4,6-DINITRO-O-CRESOL | 10 | 4.3 | 4.1 | 4.5 | 1.4 | 5.8 | 4.02 | 1.61 | 40.0 | 40 |
| PENTACHLOROPHENOL | 10 | 11 | 11 | 12 | 10 | 9.3 | 10.66 | 1.04 | 9.7 | 107 |
| 4-NITROPHENOL | 10 | (59) | 74 | 76 | 78 | 79 | 76.5 | 2.89 | 3.8 | 765 |

## TABLE IV-12. ACID EXTRACTABLE SEMIVOLATILE ORGANICS DATA—POTW RESIDUAL WASTE, SPIKE LEVEL III

| COMPOUND | ADDED | NO_1 | NO_2 | NO_3 | NO_4 | NO_5 | MEAN | S | RSD | RECOVERY |
|---|---|---|---|---|---|---|---|---|---|---|
| 2-CHLOROPHENOL | [illegible] | 16 | 15 | 16 | 12 | 8.2 | 12.64 | 2.71 | 21.6 | 63 |
| 2-NITROPHENOL | [illegible] | 12 | 15 | 15 | [illegible] | [illegible] | 11.28 | [illegible] | [illegible] | [illegible] |
| PHENOL | 20 | 18 | 16 | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] |
| 2,4-DIMETHYLPHENOL | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] |
| 2,4-DICHLOROPHENOL | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] |
| 2,4,6-TRICHLOROPHENOL | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] |
| 4-CHLORO-3-METHYLPHENOL | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] |
| 2,4-DINITROPHENOL | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] |
| 4,6-DINITRO-O-CRESOL | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] |
| PENTACHLOROPHENOL | [illegible] | [illegible] | 48 | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] |
| 4-NITROPHENOL | 20 | 65 | (65) | 87 | 87 | 86 | [illegible] | 1.50 | 1.7 | [illegible] |

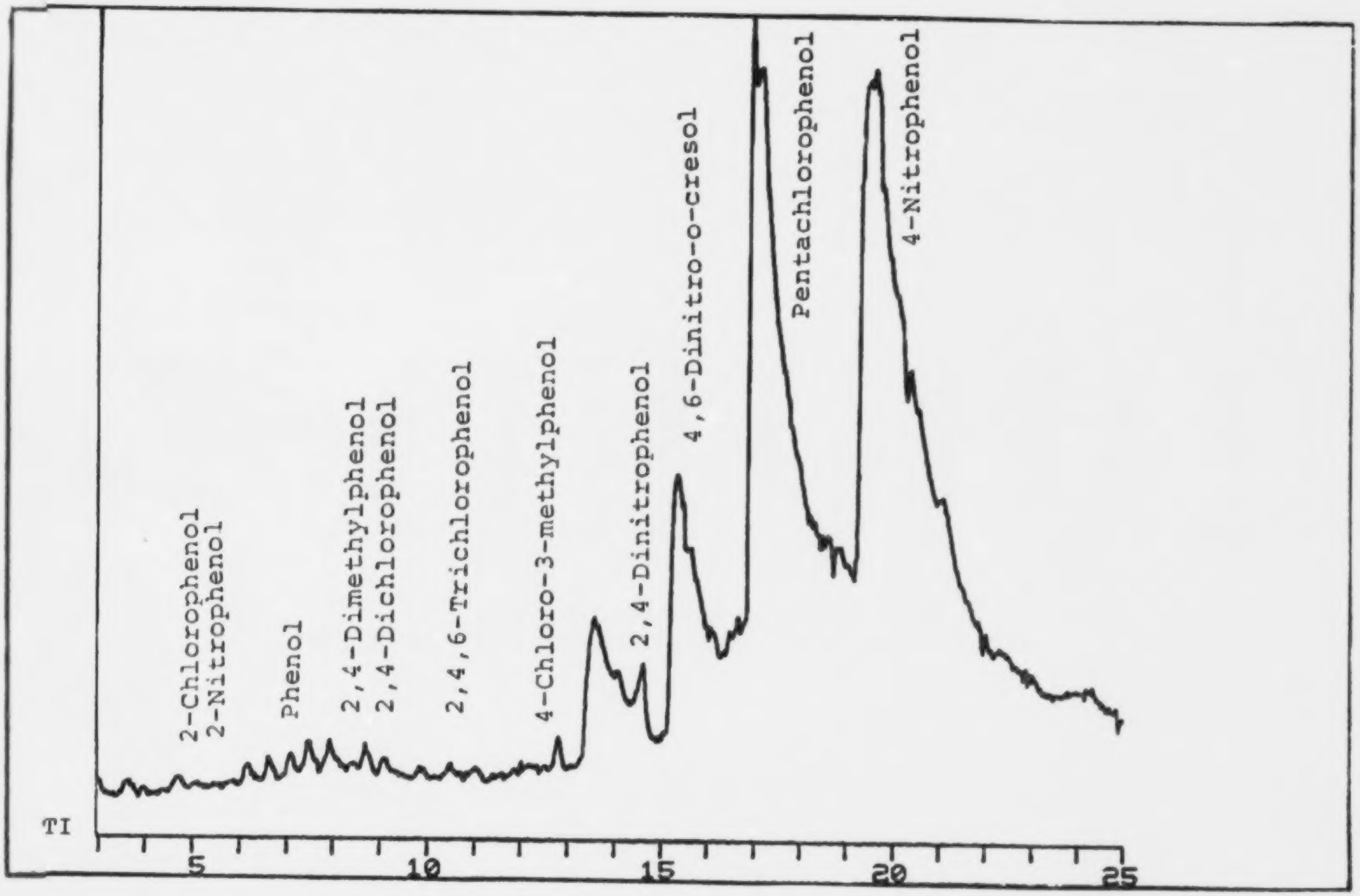


Figure IV-9. GC/MS chromatogram of acid extractables—POTW Residual Waste, spiked

## C. Ink Pigment Waste

## TABLE IV-13. BASE/NEUTRAL EXTRACTABLE SEMIVOLATILE ORGANICS DATA—INK PIGMENT WASTE, SPIKE LEVEL 0

| COMPOUND | ADDED | NO_1 | NO_2 | NO_3 | NO_4 | NO_5 | MEAN | S | RSD | RECOVERY |
|---|---|---|---|---|---|---|---|---|---|---|
| 1,3-DICHLOROBENZENE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| 1,4-DICHLOROBENZENE | 0 | 27 | 27 | 21 | 24 | 24 | 24.6 | 2.51 | 10.2 | . |
| 1,2-DICHLOROBENZENE | 0 | 34 | 32 | 25 | 27 | 28 | 29.2 | 3.70 | 12.7 | . |
| HEXACHLOROETHANE | 0 | 4.9 | 4.9 | 5.1 | 4.5 | 4.6 | 4.8 | 0.24 | 5.1 | . |
| BIS(2-CHLOROETHYL) ETHER | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| BIS(2-CHLOROISOPROPYL) ETHER | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| HEXACHLOROBUTADIENE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| NITROBENZENE | 0 | 11 | 12 | 12 | 9.2 | 9.3 | 10.7 | 1.39 | 12.9 | . |
| NAPHTHALENE | 0 | 84 | 84 | 72 | 70 | 70 | 76 | 7.35 | 9.7 | . |
| 1,2,4-TRICHLOROBENZENE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| BIS(2-CHLOROETHOXY)METHANE | 0 | 3.5 | 2.8 | 2.6 | 3 | 2.5 | 2.88 | 0.40 | 13.8 | . |
| N-NITROSODI-N-PROPYLAMINE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| HEXACHLOROCYCLOPENTADIENE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| 2-CHLORONAPHTHALENE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| ISOPHORONE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| ACENAPHTHYLENE | 0 | 11 | 12 | 9.3 | 11 | 11 | 10.86 | 0.97 | 9.0 | . |
| ACENAPHTHENE | 0 | 7.8 | 7.7 | 6.5 | 8.4 | 7.7 | 7.62 | 0.69 | 9.1 | . |
| DIMETHYL PHTHALATE | 0 | 2.1 | 2.1 | 1.7 | 2 | 1.9 | 1.96 | 0.17 | 8.5 | . |
| 2,6-DINITROTOLUENE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| FLUORENE | 0 | 22 | 22 | 18 | 21 | 18 | 20.2 | 2.05 | 10.1 | . |
| 2,4-DINITROTOLUENE | 0 | (8.8) | 4.2 | 4.3 | 3.8 | 4.1 | 4.1 | 0.22 | 5.3 | . |
| 1,2-DIPHENYLHYDRAZINE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| 4-CHLOROPHENYL PHENYL ETHER | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| DIETHYL PHTHALATE | 0 | 4.7 | 4.7 | 3.5 | 2.5 | 2.9 | 3.66 | 1.01 | 27.7 | . |
| N-NITROSODIPHENYLAMINE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| HEXACHLOROBENZENE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| 4-BROMOPHENYL PHENYL ETHER | 0 | <1 | <1 | (8.4) | 1.3 | 3.1 | <1.6 | 1.01 | 63.1 | . |
| PHENANTHRENE/ANTHRACENE | 0 | 75 | 58 | 53 | 61 | 55 | 60.4 | 8.71 | 14.4 | . |
| DI-N-BUTYL PHTHALATE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| FLUORANTHENE | 0 | 24 | 22 | 19 | 22 | 20 | 21.4 | 1.95 | 9.1 | . |
| PYRENE | 0 | 16 | 15 | 12 | 15 | 14 | 14.4 | 1.52 | 10.5 | . |
| BENZIDINE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| BUTYLBENZYL PHTHALATE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| BIS(2-ETHYLHEXYL) PHTHALATE | 0 | 100 | 102 | 105 | 93 | 100 | 100 | 4.42 | 4.4 | . |
| CHRYSENE/BENZO(A)ANTHRACENE | 0 | 33 | 29 | 23 | 26 | 30 | 28.2 | 3.83 | 13.6 | . |
| 3,3'-DICHLOROBENZIDINE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| DI-N-OCTYL PHTHALATE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| BENZO(B)/BENZO(K)FLUORANTHENES | 0 | 27 | 27 | 21 | 23 | 28 | 25.2 | 3.03 | 12.0 | . |
| BENZO(A)PYRENE | 0 | 24 | 32 | 23 | 23 | 27 | 25.8 | 3.83 | 14.9 | . |
| INDENO(1,2,3-CD)PYRENE | 0 | 10 | 11 | 9.8 | 7.8 | 15 | 10.72 | 2.66 | 24.8 | . |
| DIBENZO(AH)ANTHRACENE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| BENZO(GHI)PERYLENE | 0 | 15 | 12 | 11 | 16 | 14 | 13.6 | 2.07 | 15.2 | . |

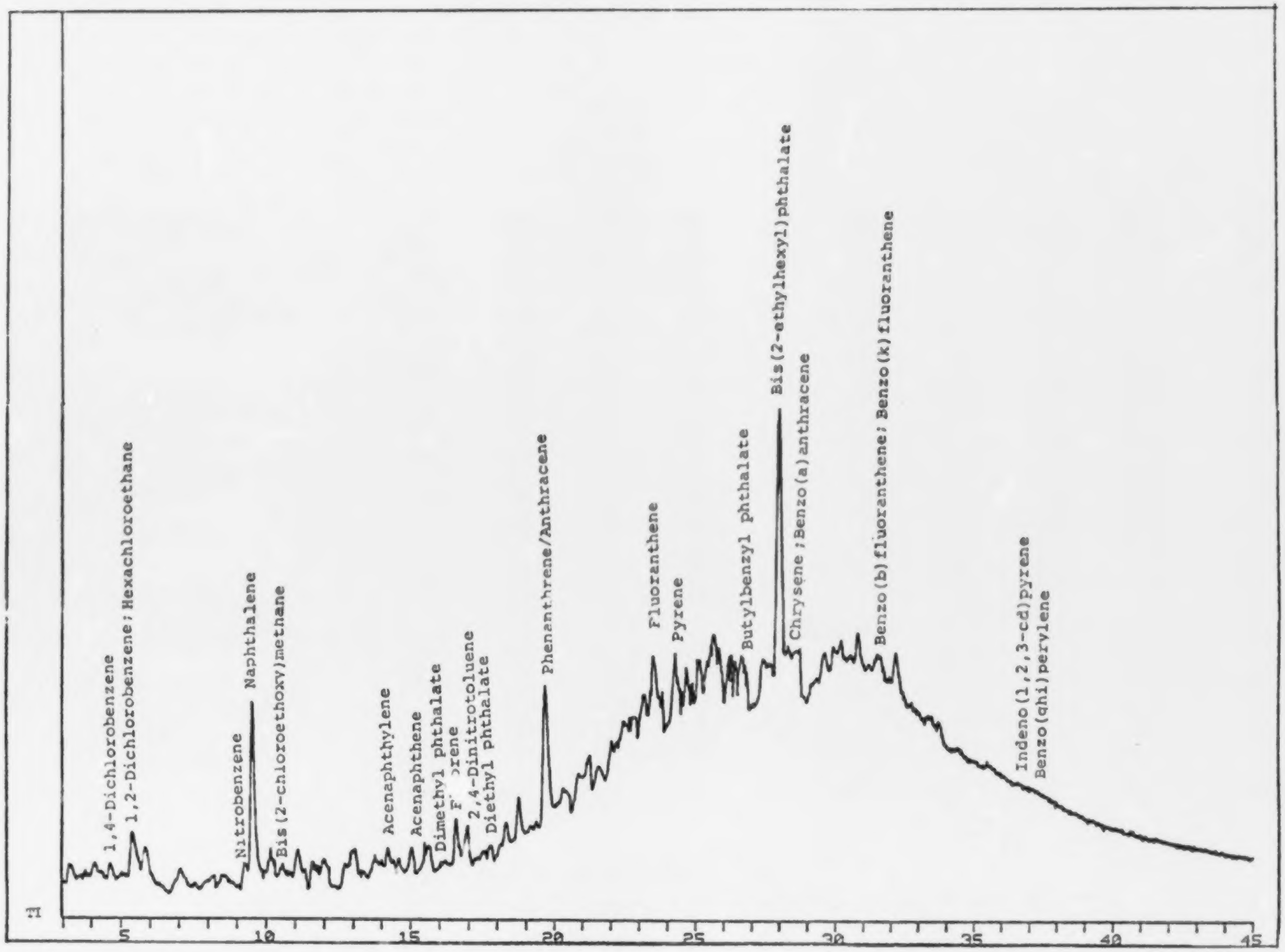


Figure IV-10. GC/MS chromatogram of base/neutral extractables—Ink Pigment Waste, unspiked

TABLE IV-14. BASE/NEUTRAL EXTRACTABLE SEMIVOLATILE ORGANICS DATA—
INK PIGMENT WASTE, SPIKE LEVEL I

| COMPOUND | ADDED | NO_1 | NO_2 | NO_3 | NO_4 | NO_5 | MEAN | S | RSD | RECOVERY |
|---|---|---|---|---|---|---|---|---|---|---|
| 1,3-DICHLOROBENZENE | 1.71 | <1 | <1 | <1 | <1 | <1 | <1 | Z | . | 58 |
| 1,4-DICHLOROBENZENE | 46.05 | 64 | 56 | 53 | 55 | 67 | 59 | 6.12 | 10.4 | 75 |
| 1,2-DICHLOROBENZENE | 77.2 | 95 | 96 | 98 | 83 | 119 | 98.2 | 13.03 | 13.3 | 89 |
| HEXACHLOROETHANE | 2 | 6.9 | 6.7 | 6.2 | 5.6 | 8 | 6.68 | 0.89 | 13.4 | 94 |
| BIS(2-CHLOROETHYL) ETHER | 2 | 2.3 | 1.6 | 2.1 | 2.6 | 1.8 | 2.08 | 0.40 | 19.0 | 104 |
| BIS(2-CHLOROISOPROPYL) ETHER | 2 | 1.7 | 2.5 | 1.9 | 2.2 | 1.7 | 2 | 0.35 | 17.3 | 100 |
| HEXACHLOROBUTADIENE | 2.05 | 2 | 1.9 | 2.2 | 2.3 | (3.3) | 2.1 | 0.18 | 8.7 | 102 |
| NITROBENZENE | 42.1 | 17 | 23 | 20 | 22 | 20 | 20.4 | 2.30 | 11.3 | 23 |
| NAPHTHALENE | 152.5 | 170 | 204 | 170 | 196 | 194 | 186.8 | 15.79 | 8.5 | 73 |
| 1,2,4-TRICHLOROBENZENE | 2.2 | 3.2 | 3.2 | 3.3 | 2.9 | 3.9 | 3.3 | 0.37 | 11.1 | 150 |
| BIS(2-CHLOROETHOXY)METHANE | 12.12 | 11 | 13 | 11 | 10 | 13 | 11.6 | 1.34 | 11.6 | 72 |
| N-NITROSODI-N-PROPYLAMINE | 2 | 11 | 14 | 11 | 14 | 16 | 13.2 | 2.17 | 16.4 | 660 |
| HEXACHLOROCYCLOPENTADIENE | 2.02 | <1 | <1 | <1 | <1 | <1 | <1 | Z | . | 50 |
| 2-CHLORONAPHTHALENE | 1.92 | 2.5 | 2.5 | 2.4 | 2.3 | 2.9 | 2.52 | 0.23 | 9.0 | 131 |
| ISOPHORONE | 2.01 | 2 | 2 | <1 | 1.4 | 1.2 | <1.52 | 0.46 | 30.3 | 76 |
| ACENAPHTHYLENE | 22.16 | 33 | 27 | 33 | 32 | 40 | 33 | 4.64 | 14.1 | 100 |
| ACENAPHTHENE | 17.11 | 24 | 23 | 23 | 23 | 31 | 24.8 | 3.49 | 14.1 | 100 |
| DIMETHYL PHTHALATE | 7.06 | 10 | 11 | 12 | 10 | 13 | 11.2 | 1.30 | 11.6 | 131 |
| 2,6-DINITROTOLUENE | 2 | 26 | 24 | 22 | 21 | 28 | 24.2 | 2.86 | 11.8 | 1210 |
| FLUORENE | 52.2 | 72 | 69 | 72 | 62 | 82 | 71.4 | 7.20 | 10.1 | 98 |
| 2,4-DINITROTOLUENE | 12.18 | 14 | 7.1 | 13 | 13 | 17 | 12.82 | 3.59 | 28.0 | 72 |
| 1,2-DIPHENYLHYDRAZINE | 2 | 1.7 | 2.2 | 1.7 | <1 | 1.4 | <1.6 | 0.44 | 27.6 | 80 |
| 4-CHLOROPHENYL PHENYL ETHER | 2.01 | 2.3 | 2.4 | 2.2 | 2.7 | 3.4 | 2.6 | 0.48 | 18.6 | 129 |
| DIETHYL PHTHALATE | 2.02 | 6.9 | 9.9 | 8.6 | 9.1 | 10 | 8.9 | 1.26 | 14.1 | 259 |
| N-NITROSODIPHENYLAMINE | 2 | 24 | 26 | 15 | 20 | 21 | 21.2 | 4.21 | 19.8 | 1060 |
| HEXACHLOROBENZENE | 1.61 | 1.3 | 1.5 | 1.5 | 1.7 | (2.7) | 1.5 | 0.16 | 10.9 | 93 |
| 4-BROMOPHENYL PHENYL ETHER | 2.07 | 3.8 | 3.2 | 2.7 | 4.5 | 4 | 3.64 | 0.70 | 19.3 | 99 |
| PHENANTHRENE/ANTHRACENE | 190.5 | 216 | 213 | 214 | 201 | 269 | 222.6 | 26.60 | 11.9 | 85 |
| DI-N-BUTYL PHTHALATE | 2.01 | 1.8 | 1.6 | <1 | 1.6 | <1 | <1.4 | 0.37 | 26.7 | 70 |
| FLUORANTHENE | 22.51 | 32 | 34 | 32 | 38 | 44 | 36 | 5.10 | 14.2 | 65 |
| PYRENE | 23.22 | 34 | 30 | 30 | 35 | 41 | 34 | 4.53 | 13.3 | 84 |
| BENZIDINE | 2 | <1 | <1 | <1 | <1 | <1 | <1 | Z | . | 50 |
| BUTYLBENZYL PHTHALATE | 2.02 | 4.9 | 5.7 | 2.8 | 2.9 | 7.4 | 4.74 | 1.95 | 41.1 | 235 |
| BIS(2-ETHYLHEXYL) PHTHALATE | 32.21 | 120 | 157 | 136 | 133 | 153 | 139.8 | 15.19 | 10.9 | 124 |
| CHRYSENE/BENZO(A)ANTHRACENE | 123.25 | 185 | 158 | 174 | 193 | 203 | 182.6 | 17.39 | 9.5 | 125 |
| 3,3'-DICHLOROBENZIDINE | 2 | <1 | <1 | <1 | <1 | <1 | <1 | Z | . | 50 |
| DI-N-OCTYL PHTHALATE | 2.03 | <1 | <1 | <1 | <1 | <1 | <1 | Z | . | 49 |
| BENZO(B)/BENZO(K)FLUORANTHENES | 124 | 231 | 189 | 206 | 213 | 289 | 225.6 | 38.51 | 17.1 | 162 |
| BENZO(A)PYRENE | 60.45 | 119 | 155 | 166 | 153 | 157 | 150 | 18.03 | 12.0 | 205 |
| INDENO(1,2,3-CD)PYRENE | 50.1 | 88 | 79 | 76 | 101 | 128 | 94.4 | 21.15 | 22.4 | 167 |
| DIBENZO(AH)ANTHRACENE | 61.4 | 120 | 94 | 92 | 128 | 155 | 117.8 | 26.10 | 22.2 | 192 |
| BENZO(GHI)PERYLENE | 62 | 133 | 108 | 136 | 140 | 160 | 135.4 | 18.60 | 13.7 | 196 |

## TABLE IV-15. BASE/NEUTRAL EXTRACTABLE SEMIVOLATILE ORGANICS DATA—INK PIGMENT WASTE, SPIKE LEVEL II

| COMPOUND | ADDED | NO_1 | NO_2 | NO_3 | NO_4 | NO_5 | MEAN | S | RSD | RECOVERY |
|---|---|---|---|---|---|---|---|---|---|---|
| 1,3-DICHLOROBENZENE | 8.54 | 6.2 | 4.3 | 5.3 | 7 | 12 | 6.96 | 2.99 | 43.0 | 81 |
| 1,4-DICHLOROBENZENE | 54.8 | 60 | 50 | 74 | 65 | 68 | 63.4 | 9.04 | 14.3 | 71 |
| 1,2-DICHLOROBENZENE | 85.2 | 109 | 89 | 120 | 109 | 103 | 106 | 11.31 | 10.7 | 95 |
| HEXACHLOROETHANE | 10.02 | 12 | 9.2 | 14 | 12 | 12 | 11.84 | 1.71 | 14.5 | 70 |
| BIS(2-CHLOROETHYL) ETHER | 10 | 13 | 11 | 17 | 11 | 14 | 13.2 | 2.49 | 18.9 | 132 |
| BIS(2-CHLOROISOPROPYL) ETHER | 10.02 | 6.6 | 3.7 | 8.2 | 8.5 | 5.8 | 6.56 | 1.95 | 29.7 | 65 |
| HEXACHLOROBUTADIENE | 10.23 | 10 | 10 | 14 | 13 | 11 | 11.6 | 1.82 | 15.7 | 113 |
| NITROBENZENE | 10.03 | 30 | 20 | 23 | 30 | 26 | 25.8 | 4.38 | 17.0 | 151 |
| NAPHTHALENE | 160.5 | 226 | 212 | 223 | 226 | 231 | 223.6 | 7.09 | 3.2 | 92 |
| 1,2,4-TRICHLOROBENZENE | 11 | 11 | 11 | 15 | 14 | 13 | 12.8 | 1.79 | 14.0 | 116 |
| BIS(2-CHLOROETHOXY)METHANE | 40.34 | 31 | 25 | 33 | 30 | 34 | 30.6 | 3.51 | 11.5 | 69 |
| N-NITROSODI-N-PROPYLAMINE | 10.02 | 16 | 12 | 13 | 16 | 15 | 14.4 | 1.82 | 12.6 | 144 |
| HEXACHLOROCYCLOPENTADIENE | 10.11 | <1 | <1 | <1 | <1 | <1 | <1 | 2 | . | 10 |
| 2-CHLORONAPHTHALENE | 9.6 | 11 | 8.2 | 13 | 11 | 12 | 11.04 | 1.79 | 16.2 | 115 |
| ISOPHORONE | 10.08 | 5.9 | 3.8 | 6 | 4.7 | 5.9 | 5.26 | 0.98 | 18.6 | 52 |
| ACENAPHTHYLENE | 110.8 | 122 | 99 | 134 | 121 | 124 | 120 | 12.83 | 10.7 | 99 |
| ACENAPHTHENE | 85.55 | 86 | 70 | 97 | 86 | 86 | 85 | 9.64 | 11.3 | 90 |
| DIMETHYL PHTHALATE | 35.21 | 18 | 16 | 23 | 21 | 17 | 19 | 2.92 | 15.3 | 48 |
| 2,6-DINITROTOLUENE | 10.05 | 27 | 26 | 32 | 34 | 33 | 30.4 | 3.65 | 12.0 | 302 |
| FLUORENE | 60.25 | 74 | 57 | 90 | 85 | 83 | 77.8 | 12.99 | 16.7 | 96 |
| 2,4-DINITROTOLUENE | 60.95 | 57 | 46 | 63 | 62 | 53 | 56.2 | 6.98 | 12.4 | 85 |
| 1,2-DIPHENYLHYDRAZINE | 10.02 | 4.7 | 4.4 | 5.9 | 5.1 | 6.4 | 5.3 | 0.83 | 15.7 | 53 |
| 4-CHLOROPHENYL PHENYL ETHER | 10.04 | 12 | 9.2 | 13 | 13 | 11 | 11.64 | 1.60 | 13.7 | 116 |
| DIETHYL PHTHALATE | 10.11 | 16 | 11 | 6.9 | 13 | 12 | 11.78 | 3.31 | 28.1 | 80 |
| N-NITROSODIPHENYLAMINE | 10 | 15 | 18 | 27 | 21 | 44 | 25 | 11.51 | 46.0 | 250 |
| HEXACHLOROBENZENE | 8.06 | 7.2 | 6.6 | 11 | 9.3 | 8 | 8.38 | 1.81 | 21.7 | 104 |
| 4-BROMOPHENYL PHENYL ETHER | 10.34 | 12 | 10 | 18 | 15 | 13 | 13.6 | 3.05 | 22.4 | 116 |
| PHENANTHRENE/ANTHRACENE | 206.7 | 225 | 211 | 261 | 240 | 232 | 233.8 | 18.57 | 7.9 | 84 |
| DI-N-BUTYL PHTHALATE | 10.36 | 9.9 | 8.3 | 7.4 | 9.5 | 10 | 9.02 | 1.13 | 12.5 | 90 |
| FLUORANTHENE | 112.45 | 126 | 97 | 138 | 114 | 107 | 116.4 | 16.04 | 13.8 | 84 |
| PYRENE | 116.05 | 116 | 87 | 112 | 98 | 110 | 104.6 | 11.91 | 11.4 | 78 |
| BENZIDINE | 10 | 2.5 | 1.6 | 2.1 | 1.4 | 1.2 | 1.76 | 0.53 | 30.2 | 18 |
| BUTYLBENZYL PHTHALATE | 10.09 | 6 | 3.6 | 6.9 | 8.2 | 16 | 8.14 | 4.70 | 57.8 | 81 |
| BIS(2-ETHYLHEXYL) PHTHALATE | 161.15 | 301 | 282 | 364 | 305 | 369 | 324.2 | 39.62 | 12.2 | 139 |
| CHRYSENE/BENZO(A)ANTHRACENE | 123.25 | 209 | 163 | 225 | 161 | 90 | 169.6 | 52.62 | 31.0 | 115 |
| 3,3'-DICHLOROBENZIDINE | 10 | 4.5 | 2.4 | 6.7 | 2.5 | 7.2 | 4.66 | 2.26 | 48.5 | 47 |
| DI-N-OCTYL PHTHALATE | 10.13 | <1 | <1 | <1 | <1 | <1 | <1 | 2 | . | 10 |
| BENZO(B)/BENZO(K)FLUORANTHENES | 124 | 214 | 186 | 265 | 194 | 248 | 221.4 | 34.16 | 15.4 | 158 |
| BENZO(A)PYRENE | 60.95 | 195 | 166 | 140 | 192 | 230 | 184.6 | 33.76 | 18.3 | 261 |
| INDENO(1,2,3-CD)PYRENE | 50.1 | 85 | 92 | 111 | 72 | 96 | 91.2 | 14.34 | 15.7 | 161 |
| DIBENZO(AH)ANTHRACENE | 61.4 | 107 | 114 | 142 | 100 | 139 | 120.4 | 19.03 | 15.8 | 196 |
| BENZO(GHI)PERYLENE | 62 | 131 | 104 | 139 | 90 | 115 | 115.8 | 19.84 | 17.1 | 165 |

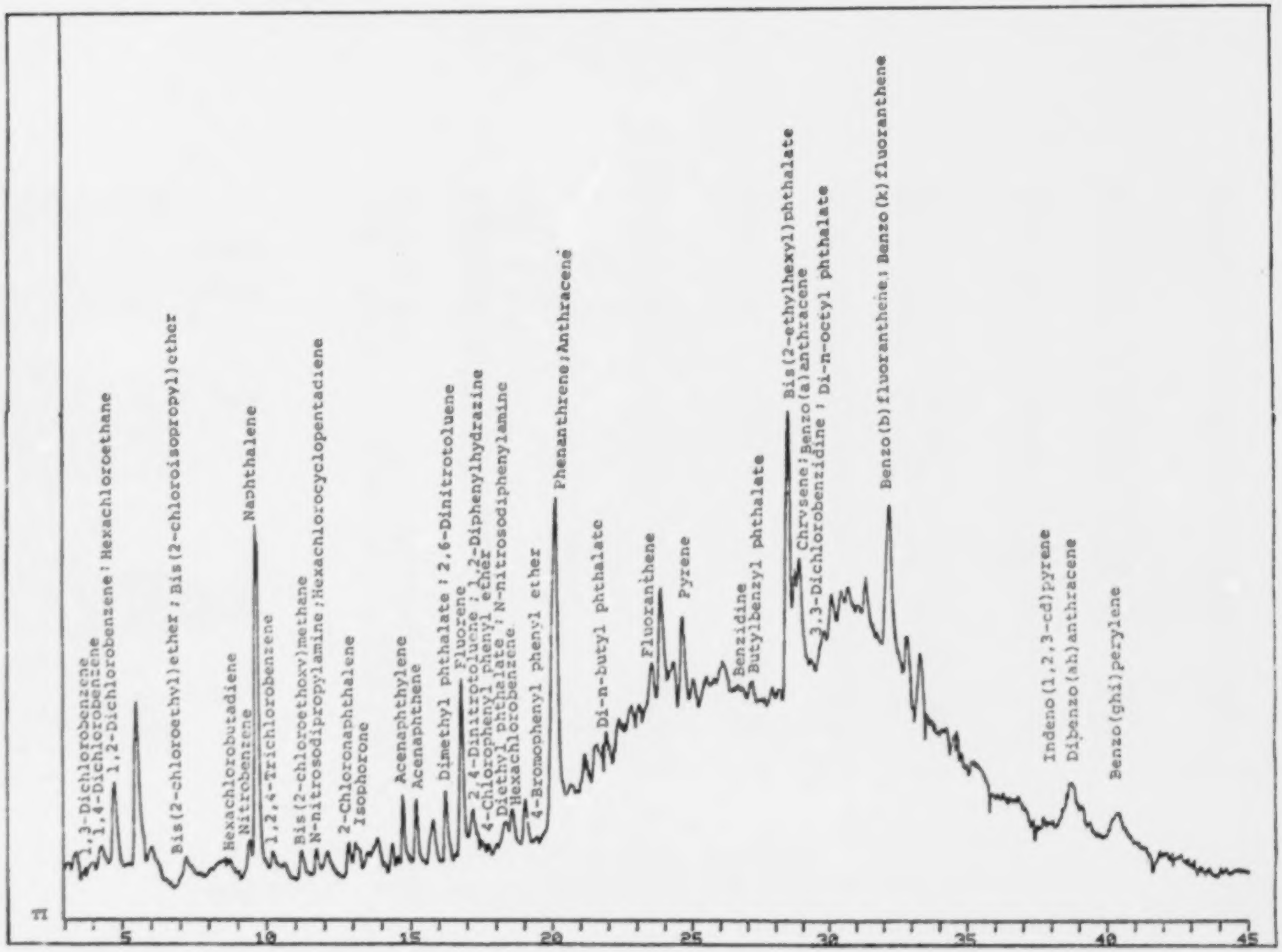


Figure IV-11. GC/MS chromatogram of base/neutral extractables—
Ink Pigment Waste, spiked

TABLE IV-16. ACID EXTRACTABLE SEMIVOLATILE ORGANICS DATA—
INK PIGMENT WASTE, SPIKE LEVEL 0

| COMPOUND | ADDED | NO_1 | NO_2 | NO_3 | NO_4 | NO_5 | MEAN | S | RSD | RECOVERY |
|---|---|---|---|---|---|---|---|---|---|---|
| 2-CHLOROPHENOL | 0 | 4.9 | 2.3 | 1.9 | 3.2 | 5.6 | 3.58 | 1.61 | 45.1 | . |
| 2-NITROPHENOL | 0 | <1 | <1 | <1 | <1 | <1 | <1 | Z | . | . |
| PHENOL | 0 | 54 | 49 | 57 | 62 | 55 | 55.4 | 4.72 | 8.5 | . |
| 2,4-DIMETHYLPHENOL | 0 | 1 | 1 | <1 | 1.2 | 1.6 | <1.16 | 0.26 | 22.5 | . |
| 2,4-DICHLOROPHENOL | 0 | <1 | 3 | <1 | 0 | 0 | <0.4 | Z | . | . |
| 2,4,6-TRICHLOROPHENOL | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| 4-CHLORO-3-METHYLPHENOL | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| 2,4-DINITROPHENOL | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| 4,6-DINITRO-O-CRESOL | 0 | 0 | 0 | 0 | <1 | <1 | <0.4 | Z | . | . |
| PENTACHLOROPHENOL | 0 | 1.4 | 3 | 1.5 | 2.9 | 3.6 | 2.48 | 0.98 | 39.4 | . |
| 4-NITROPHENOL | 0 | 5.2 | 6.7 | 4.5 | 6.2 | 7.9 | 6.1 | 1.32 | 21.7 | . |

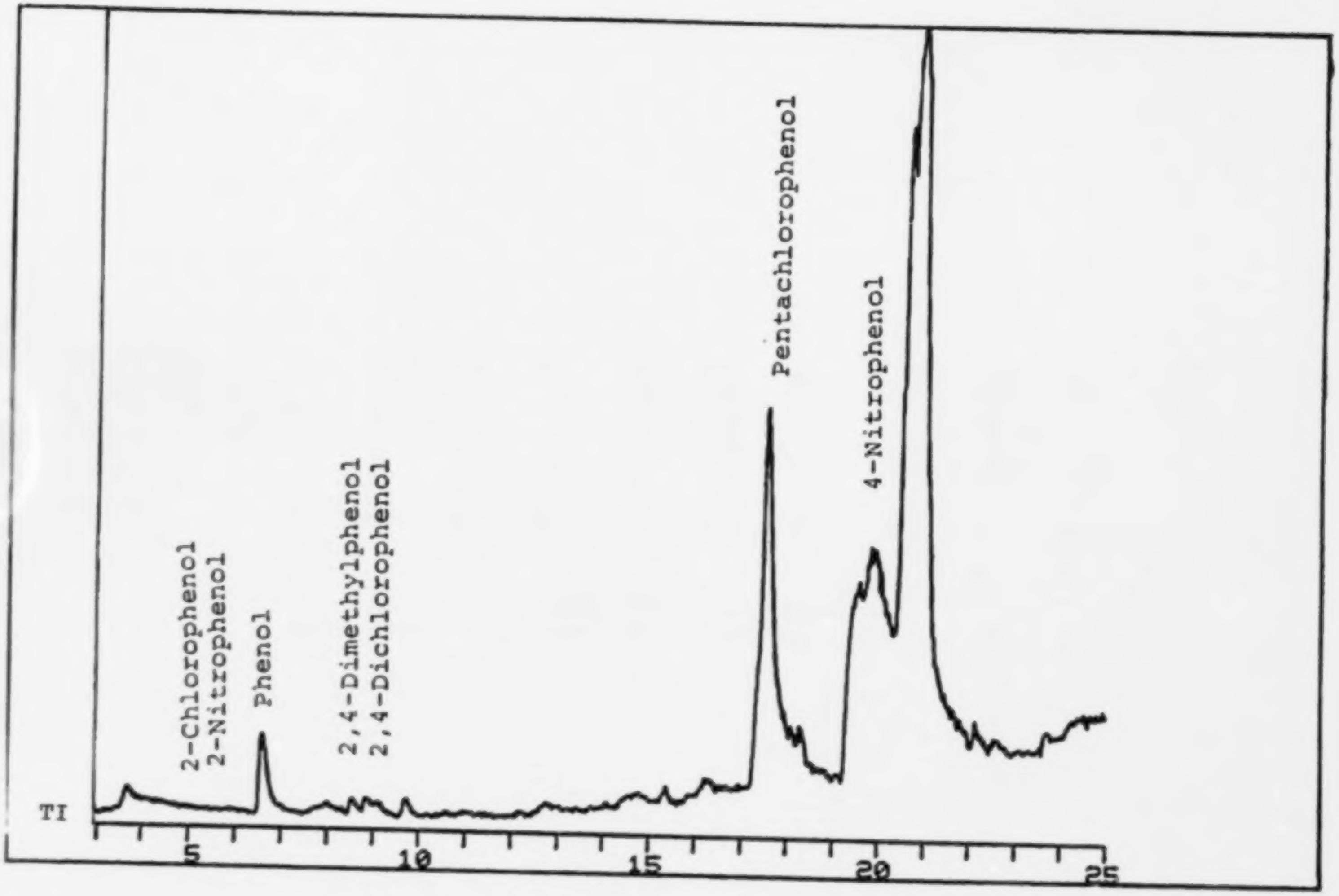


Figure IV-12. GC/MS chromatogram of acid extractables—Ink Pigment Waste, unspiked

## TABLE IV-17. ACID EXTRACTABLE SEMIVOLATILE ORGANICS DATA— INK PIGMENT WASTE, SPIKE LEVEL I

| COMPOUND | ADDED | NO_1 | NO_2 | NO_3 | NO_4 | NO_5 | MEAN | S | RSD | RECOVERY |
|---|---|---|---|---|---|---|---|---|---|---|
| 2-CHLOROPHENOL | 26.45 | 35 | 27 | 32 | 26 | 45 | 33 | 7.65 | 23.2 | 111 |
| 2-NITROPHENOL | 3 | 3.6 | 3.1 | 3.7 | 2 | 3.9 | 3.26 | 0.76 | 23.4 | 113 |
| PHENOL | 123.25 | 153 | 106 | 133 | 106 | 203 | 140.2 | 40.31 | 28.8 | 69 |
| 2,4-DIMETHYLPHENOL | 6.26 | 2.3 | 2.5 | 3 | 2.1 | (5.0) | 2.475 | 0.39 | 15.6 | 21 |
| 2,4-DICHLOROPHENOL | 2.02 | 1.1 | 1 | 1.3 | 0.7 | (2.4) | 1.025 | 0.25 | 24.4 | 31 |
| 2,4,6-TRICHLOROPHENOL | 2.01 | 1.6 | 0.7 | 1 | 0.6 | 2.5 | 1.28 | 0.79 | 61.4 | 64 |
| 4-CHLORO-3-METHYLPHENOL | 2 | 1.3 | 1.3 | 1.1 | 0.8 | 2 | 1.3 | 0.44 | 34.0 | 65 |
| 2,4-DINITROPHENOL | 2 | 3.6 | 0.9 | 1.1 | 1.7 | <0.5 | <1.56 | 1.22 | 78.2 | 78 |
| 4,6-DINITRO-O-CRESOL | 2.01 | 1.6 | 0.8 | 1.7 | 0.5 | 0.5 | 1.02 | 0.59 | 57.8 | 31 |
| PENTACHLOROPHENOL | 17.2 | 31 | 9.1 | 29 | 22 | 27 | 23.62 | 8.78 | 37.2 | 123 |
| 4-NITROPHENOL | 62.35 | 77 | 55 | 88 | 62 | 104 | 77.2 | 19.74 | 25.6 | 114 |

## TABLE IV-18. ACID EXTRACTABLE SEMIVOLATILE ORGANICS DATA— INK PIGMENT WASTE, SPIKE LEVEL II

| COMPOUND | ADDED | NO_1 | NO_2 | NO_3 | NO_4 | NO_5 | MEAN | S | RSD | RECOVERY |
|---|---|---|---|---|---|---|---|---|---|---|
| 2-CHLORLPHENOL | 35.38 | 24 | 23 | 23 | 27 | 33 | 26 | 4.24 | 16.3 | 74 |
| 2-NITROPHENOL | 10.05 | 8.4 | 7.7 | 8.8 | 11 | 13 | 9.78 | 2.18 | 22.3 | 97 |
| PHENOL | 211.23 | 129 | 140 | 150 | 156 | 167 | 148.4 | 14.60 | 9.8 | 71 |
| 2,4-DIMETHYLPHENOL | 36.34 | 10 | 7.2 | 7.6 | 9.7 | 9.1 | 8.72 | 1.26 | 14.4 | 24 |
| 2,4-DICHLOROPHENOL | 10.1 | 8 | 5.8 | 5.3 | 7.7 | 6.2 | 6.6 | 1.19 | 18.0 | 61 |
| 2,4,6-TRICHLOROPHENOL | 10.08 | 14 | 26 | 17 | 14 | 21 | 18.4 | 5.13 | 27.9 | 183 |
| 4-CHLORO-3-METHYLPHENOL | 10 | 13 | 19 | 16 | 15 | 17 | 16 | 4.93 | 30.8 | 160 |
| 2,4-DINITROPHENOL | 10 | 24 | 6.7 | 3 | 7.6 | 12 | 10.66 | 8.43 | 79.1 | 107 |
| 4,6-DINITRO-O-CRESOL | 10.04 | 2.8 | 4.2 | 2.57 | 3.9 | (14) | 3.623 | 0.98 | 27.2 | 32 |
| PENTACHLOROPHENOL | 88 | 95 | 140 | 137 | 138 | 109 | 123.8 | 50.06 | 40.4 | 141 |
| 4-NITROPHENOL | 76.35 | 75 | 63 | 18 | 87 | 117 | 72 | 36.26 | 50.3 | 94 |

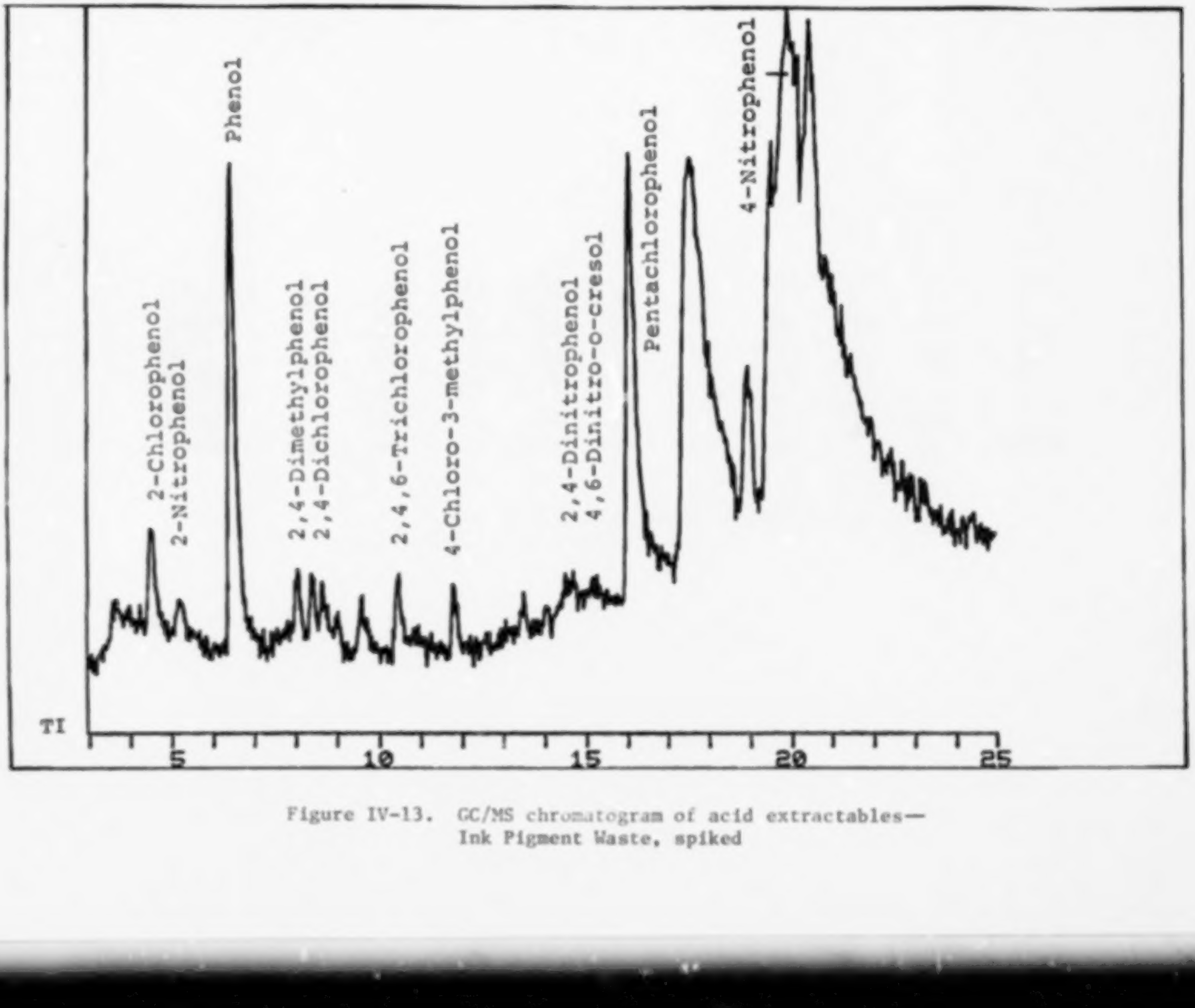


Figure IV-13. GC/MS chromatogram of acid extractables—
Ink Pigment Waste, spiked

### TABLE IV-19. METALS DATA— INK PIGMENT WASTE, SPIKE LEVEL 0

| ELEMENT | ADDED | NO_1 | NO_2 | NO_3 | NO_4 | NO_5 | MEAN | S | RSD | RECOVERY |
|---|---|---|---|---|---|---|---|---|---|---|
| ANTIMONY | 0 | 0.4 | 0.2 | 0.3 | 0.3 | 0.3 | 0.3 | 0.07 | 23.6 | . |
| ARSENIC | 0 | 0.9 | 1 | 1 | 0.8 | 0.9 | 0.92 | 0.08 | 9.1 | . |
| BERYLLIUM | 0 | <0.1 | <0.1 | <0.1 | <0.1 | <0.1 | <0.1 | . | . | . |
| CADMIUM | 0 | <0.1 | <0.1 | <0.1 | <0.1 | <0.1 | <0.1 | . | . | . |
| CHROMIUM | 0 | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | 7.8 | 0.45 | 5.8 | . |
| COPPER | 0 | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | 0.41 | 3.9 | . |
| LEAD | 0 | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | 0.10 | 2.0 | . |
| MERCURY | 0 | <0.1 | <0.1 | <0.1 | <0.1 | <0.1 | <0.1 | . | . | . |
| NICKEL | 0 | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | 0.08 | 8.2 | . |
| SELENIUM | 0 | <0.1 | <0.1 | <0.1 | <0.1 | <0.1 | <0.1 | . | . | . |
| SILVER | 0 | <0.1 | <0.1 | <0.1 | <0.1 | <0.1 | <0.1 | . | . | . |
| THALLIUM | 0 | <0.1 | <0.1 | <0.1 | <0.1 | <0.1 | <0.1 | . | . | . |
| ZINC | 0 | 30 | 30 | 29 | 29 | 29 | 29.4 | 0.55 | 1.9 | . |

## TABLE IV-20. METALS DATA—
## INK PIGMENT WASTE, SPIKE LEVEL I

| ELEMENT | ADDED | NO_1 | NO_2 | NO_3 | NO_4 | NO_5 | MEAN | S | RSD | RECOVERY |
|---|---|---|---|---|---|---|---|---|---|---|
| ANTIMONY | 6.1 | 5.3 | 5.8 | 5.2 | 5.1 | 4.8 | 5.24 | 0.36 | 7.0 | 81 |
| ARSENIC | 6.1 | 6.2 | 7.4 | 7.6 | 7.5 | 7.6 | 7.26 | [illegible] | [illegible] | [illegible] |
| BERYLLIUM | 6.1 | 6.3 | 6.3 | [illegible] | 6.5 | 6.6 | [illegible] | [illegible] | [illegible] | [illegible] |
| CADMIUM | 6.1 | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] |
| CHROMIUM | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] |
| COPPER | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] |
| LEAD | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] |
| MERCURY | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] |
| NICKEL | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] |
| SELENIUM | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] |
| SILVER | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] |
| THALLIUM | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] | [illegible] |
| ZINC | 26.5 | 52.7 | 50.9 | 56 | 56 | 55.1 | 54.14 | 1.25 | 2.3 | 103 |

## D. Organic Still Bottoms

TABLE IV-21. BASE/NEUTRAL EXTRACTABLE SEMIVOLATILE ORGANICS DATA—
ORGANIC STILL BOTTOMS, SPIKE LEVEL 0

| COMPOUND | ADDED | NO_1 | NO_2 | NO_3 | NO_4 | NO_5 | MEAN | S | RSD | RECOVERY |
|---|---|---|---|---|---|---|---|---|---|---|
| 1,3-DICHLOROBENZENE | 0 | 16 | 25 | 19 | 14 | 13 | 17.4 | 4.83 | 27.7 | . |
| 1,4-DICHLOROBENZENE | 0 | 104 | 131 | 135 | (247) | 88 | 114.5 | 22.40 | 19.6 | . |
| 1,2-DICHLOROBENZENE | 0 | 33 | 25 | 59 | (123) | 43 | 40 | 14.65 | 36.6 | . |
| HEXACHLOROETHANE | 0 | 654 | 631 | 665 | (1226) | 538 | 622 | 57.76 | 9.3 | . |
| BIS(2-CHLOROETHYL) ETHER | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| BIS(2-CHLOROISOPROPYL) ETHER | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| HEXACHLOROBUTADIENE | 0 | 1510 | 2420 | 3190 | (7240) | 2010 | 2282.5 | 710.28 | 31.1 | . |
| NITROBENZENE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| NAPHTHALENE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| 1,2,4-TRICHLOROBENZENE | 0 | 21 | 23 | 34 | (78) | 21 | 24.75 | 6.24 | 25.2 | . |
| BIS(2-CHLOROETHOXY)METHANE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| N-NITROSODI-N-PROPYLAMINE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| HEXACHLOROCYCLOPENTADIENE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| 2-CHLORONAPHTHALENE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| ISOPHORONE | 0 | 25 | 22 | 30 | (52) | 21 | 24.5 | 4.04 | 16.5 | . |
| ACENAPHTHYLENE | 0 | 324 | 349 | 492 | (955) | 273 | 359.5 | 93.82 | 26.1 | . |
| ACENAPHTHENE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| DIMETHYL PHTHALATE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| 2,6-DINITROTOLUENE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| FLUORENE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| 2,4-DINITROTOLUENE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| 1,2-DIPHENYLHYDRAZINE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| 4-CHLOROPHENYL PHENYL ETHER | 0 | 5.1 | 2.7 | 3.9 | 4.5 | 2.7 | 3.78 | 1.07 | 28.4 | . |
| DIETHYL PHTHALATE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| N-NITROSODIPHENYLAMINE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| HEXACHLOROBENZENE | 0 | 35 | 45 | 70 | 83 | 72 | 61 | 20.11 | 33.0 | . |
| 4-BROMOPHENYL PHENYL ETHER | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| PHENANTHRENE/ANTHRACENE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| DI-N-BUTYL PHTHALATE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| FLUORANTHENE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| PYRENE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| BENZIDINE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| BUTYLBENZYL PHTHALATE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| BIS(2-ETHYLHEXYL) PHTHALATE | 0 | 0.85 | 0.82 | 1.3 | 1.9 | 0.62 | 1.098 | 0.51 | 46.7 | . |
| CHRYSENE/BENZO(A)ANTHRACENE | 0 | 1 | 1.2 | 3.5 | 4.6 | 1.3 | 2.32 | 1.63 | 70.3 | . |
| 3,3'-DICHLOROBENZIDINE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| DI-N-OCTYL PHTHALATE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| BENZO(B)/BENZO(K)FLUORANTHENES | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| BENZO(A)PYRENE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| INDENO(1,2,3-CD)PYRENE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| DIBENZO(AH)ANTHRACENE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| BENZO(GHI)PERYLENE | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |

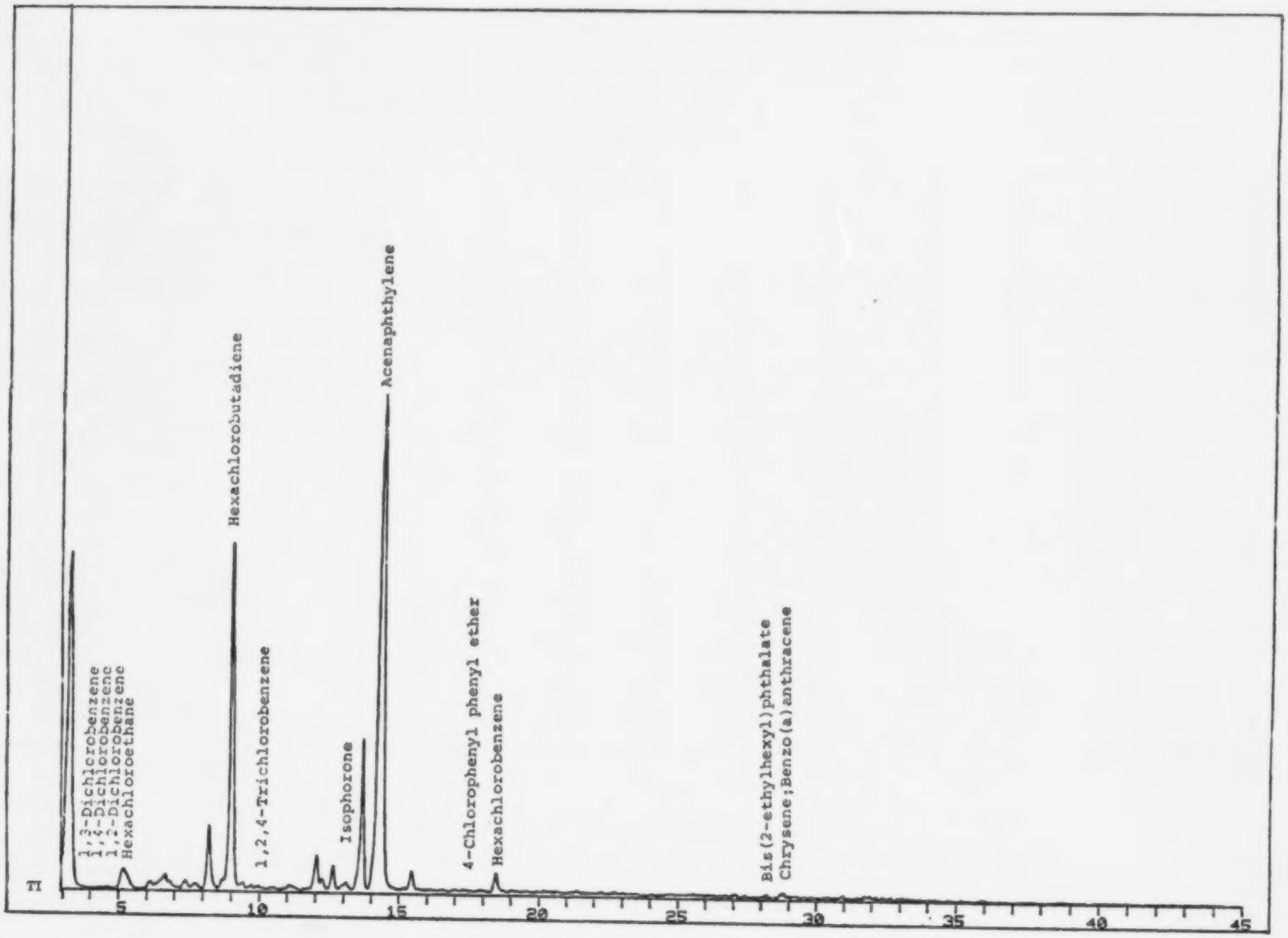


Figure IV-14. GC/MS chromatogram of base/neutral extractables — Organic Still Bottoms, unspiked

## TABLE IV-22. ACID EXTRACTABLE SEMIVOLATILE ORGANICS DATA— ORGANIC STILL BOTTOMS, SPIKE LEVEL 0

| COMPOUND | ADDED | NO_1 | NO_2 | NO_3 | NO_4 | NO_5 | MEAN | S | RSD | RECOVERY |
|---|---|---|---|---|---|---|---|---|---|---|
| 2-CHLOROPHENOL | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| 2-NITROPHENOL | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| PHENOL | 0 | 4.3 | 5.5 | 4.9 | 3.5 | 4.1 | 4.46 | 0.77 | 17.2 | . |
| 2,4-DIMETHYLPHENOL | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| 2,4-DICHLOROPHENOL | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| 2,4,6-TRICHLOROPHENOL | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| 4-CHLORO-3-METHYLPHENOL | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| 2,4-DINITROPHENOL | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| 4,6-DINITRO-O-CRESOL | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| PENTACHLOROPHENOL | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |
| 4-NITROPHENOL | 0 | 0 | 0 | 0 | 0 | 0 | 0 | Z | . | . |

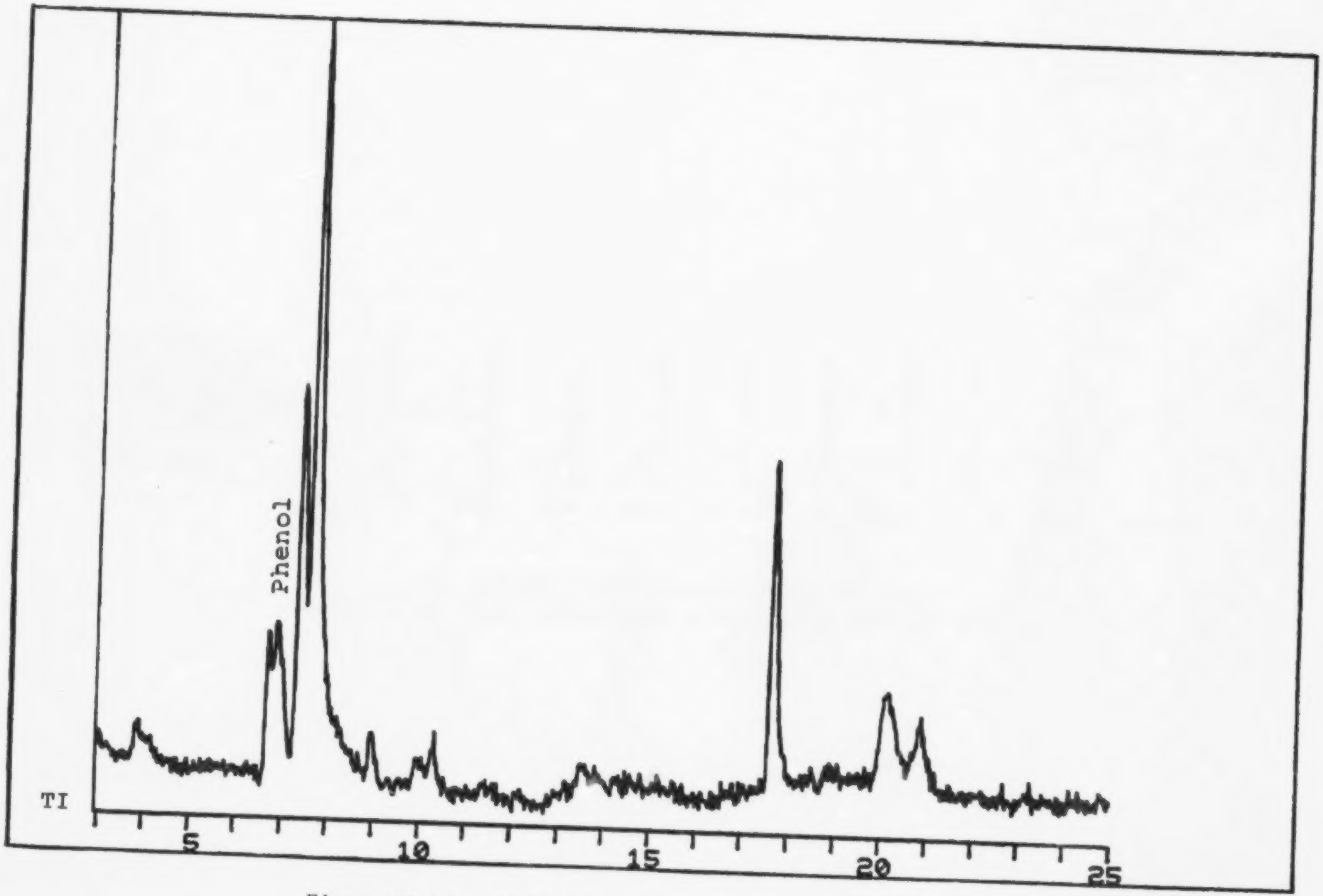


Figure IV-15. GC/MS chromatogram of acid extractables—
Organic Still Bottoms, unspiked

TABLE IV-23. METALS DATA—
ORGANIC STILL BOTTOMS, SPIKE LEVEL 0

| ELEMENT | ADDED | NO_1 | NO_2 | NO_3 | NO_4 | NO_5 | MEAN | S | RSD | RECOVERY |
|---|---|---|---|---|---|---|---|---|---|---|
| ANTIMONY | 0 | <0.1 | <0.1 | <0.1 | <0.1 | <0.1 | <0.1 | Z | . | . |
| ARSENIC | 0 | 0.9 | 1.1 | 0.9 | 1.1 | 1 | 1 | 0.10 | 10.0 | . |
| BERYLLIUM | 0 | <0.1 | <0.1 | <0.1 | <0.1 | <0.1 | <0.1 | Z | . | . |
| CADMIUM | 0 | <0.2 | <0.2 | <0.2 | <0.2 | <0.2 | <0.2 | Z | . | . |
| CHROMIUM | 0 | 38 | 36 | 36 | 40 | 35 | 37 | 2.00 | 5.4 | . |
| COPPER | 0 | 636 | 621 | 625 | 633 | 622 | 627.4 | 6.73 | 1.1 | . |
| LEAD | 0 | 11 | 10 | 11 | 11 | 9.7 | 10.54 | 0.64 | 6.1 | . |
| MERCURY | 0 | 0.9 | 0.8 | 0.8 | 0.6 | 0.7 | 0.76 | 0.11 | 15.0 | . |
| NICKEL | 0 | 103 | 106 | 106 | 102 | 103 | 104 | 1.87 | 1.8 | . |
| SELENIUM | 0 | <0.7 | <0.7 | <0.7 | <0.7 | <0.7 | <0.7 | Z | . | . |
| SILVER | 0 | 0.4 | 0.3 | 0.2 | 0.2 | 0.2 | 0.26 | 0.09 | 34.4 | . |
| THALLIUM | 0 | <0.3 | <0.3 | <0.3 | <0.3 | <0.3 | <0.3 | Z | . | . |
| ZINC | 0 | 54 | 55 | 55 | 57 | . | 55.25 | 1.26 | 2.3 | . |

## TABLE IV-24. METALS DATA— ORGANIC STILL BOTTOMS, SPIKE LEVEL I

| ELEMENT | ADDED | NO_1 | NO_2 | NO_3 | NO_4 | NO_5 | MEAN | S | RSD | RECOVERY |
|---|---|---|---|---|---|---|---|---|---|---|
| ANTIMONY | 5 | 0.2 | 0.2 | 0.1 | 0.12 | 0.3 | 0.184 | 0.08 | 43.1 | 2 |
| ARSENIC | 5 | 4.7 | 6.2 | 5 | 5.5 | 6.52 | 5.584 | 0.77 | 13.8 | 92 |
| BERYLLIUM | 5 | 3.6 | 4.8 | 3.8 | 4.2 | 5.03 | 4.286 | 0.62 | 14.4 | 84 |
| CADMIUM | 5 | 4.12 | 5.6 | 4.4 | 4.8 | 5.31 | 4.846 | 0.61 | 12.7 | 93 |
| CHROMIUM | 20 | 51 | 61 | 53 | 58 | 61 | 56.8 | 4.60 | 8.1 | 99 |
| COPPER | 500 | 1071 | 1194 | 902 | 1037 | 1104 | 1061.6 | 106.65 | 10.0 | 87 |
| LEAD | 10 | 18 | 22 | 19 | 21 | 22 | 20.4 | 1.82 | 8.9 | 99 |
| MERCURY | 5 | 3.7 | 4.9 | 4.4 | 4.9 | 4.5 | 4.48 | 0.49 | 11.0 | 74 |
| NICKEL | 200 | 263 | 318 | 266 | 286 | 312 | 289 | 25.42 | 8.8 | 92 |
| SELENIUM | 5 | 2.4 | 3.6 | 2.3 | 3.2 | 3.5 | 3 | 0.61 | 20.4 | 46 |
| SILVER | 5 | 4.3 | 5.7 | 4.3 | 5.1 | 5.8 | 5.04 | 0.73 | 14.4 | 96 |
| THALLIUM | 5 | 2 | 2.6 | 2.1 | 2.3 | 3.2 | 2.44 | 0.48 | 19.8 | 43 |
| ZINC | 50 | 98 | 116 | 95 | 90 | 111 | 102 | 11.02 | 10.8 | 93 |

## E. Paint Pigment Sludge

TABLE IV-25. PURGEABLE ORGANICS DATA—
PAINT PIGMENT SLUDGE, SPIKE LEVEL 0

| COMPOUND | ADDED | NO_1 | NO_2 | NO_3 | NO_4 | NO_5 | MEAN | S | RSD | RECOVERY |
|---|---|---|---|---|---|---|---|---|---|---|
| METHYLENE CHLORIDE | 0 | 0.65 | 0.67 | 0.91 | . | . | 0.743333 | 0.14 | 19.5 | . |
| 1,1-DICHLOROETHYLENE | 0 | 0 | 0 | 0 | . | . | 0 | Z | . | . |
| 1,1-DICHLOROETHANE | 0 | 0 | 0 | 0 | . | . | 0 | Z | . | . |
| TRANS-1,2-DICHLOROETHYLENE | 0 | 0 | 0 | 0 | . | . | 0 | Z | . | . |
| CHLOROFORM | 0 | 0 | 0 | 0 | . | . | 0 | Z | . | . |
| 1,2-DICHLOROETHANE | 0 | 0 | 0 | 0 | . | . | 0 | Z | . | . |
| 1,1,1-TRICHLOROETHANE | 0 | 0 | 0 | 0 | . | . | 0 | Z | . | . |
| CARBON TETRACHLORIDE | 0 | 0 | 0 | 0 | . | . | 0 | Z | . | . |
| BROMODICHLOROMETHANE | 0 | 0 | 0 | 0 | . | . | 0 | Z | . | . |
| 1,2-DICHLOROPROPANE | 0 | 0 | 0 | 0 | . | . | 0 | Z | . | . |
| TRANS-1,3-DICHLOROPROPENE | 0 | 0 | 0 | 0 | . | . | 0 | Z | . | . |
| TRICHLOROETHYLENE | 0 | 2.2 | 1.1 | 1.9 | . | . | 1.73333 | 0.57 | 32.8 | . |
| DIBROMOCHLOROMETHANE | 0 | 0 | 0 | 0 | . | . | 0 | Z | . | . |
| CIS-1,3-DICHLOROPROPENE | 0 | 0 | 0 | 0 | . | . | 0 | Z | . | . |
| 1,1,2-TRICHLOROETHANE | 0 | 0 | 0 | 0 | . | . | 0 | Z | . | . |
| BENZENE | 0 | 4.6 | 3.2 | 3.7 | . | . | 3.83333 | 0.71 | 18.5 | . |
| BROMOFORM | 0 | 0 | 0 | 0 | . | . | 0 | Z | . | . |
| 1,1,2,2-TETRACHLOROETHENE | 0 | 8.1 | 6.1 | 8.2 | . | . | 7.46667 | 1.18 | 15.9 | . |
| 1,1,2,2-TETRACHLOROETHANE | 0 | 4 | 2.9 | 3.5 | . | . | 3.46667 | 0.55 | 15.9 | . |
| TOLUENE | 0 | 15 | 11 | 16 | . | . | 14 | 2.65 | 18.9 | . |
| CHLOROBENZENE | 0 | 2.4 | 1.6 | 2.2 | . | . | 2.06667 | 0.42 | 20.1 | . |
| ETHYLBENZENE | 0 | 261 | 200 | 291 | . | . | 250.667 | 46.37 | 18.5 | . |

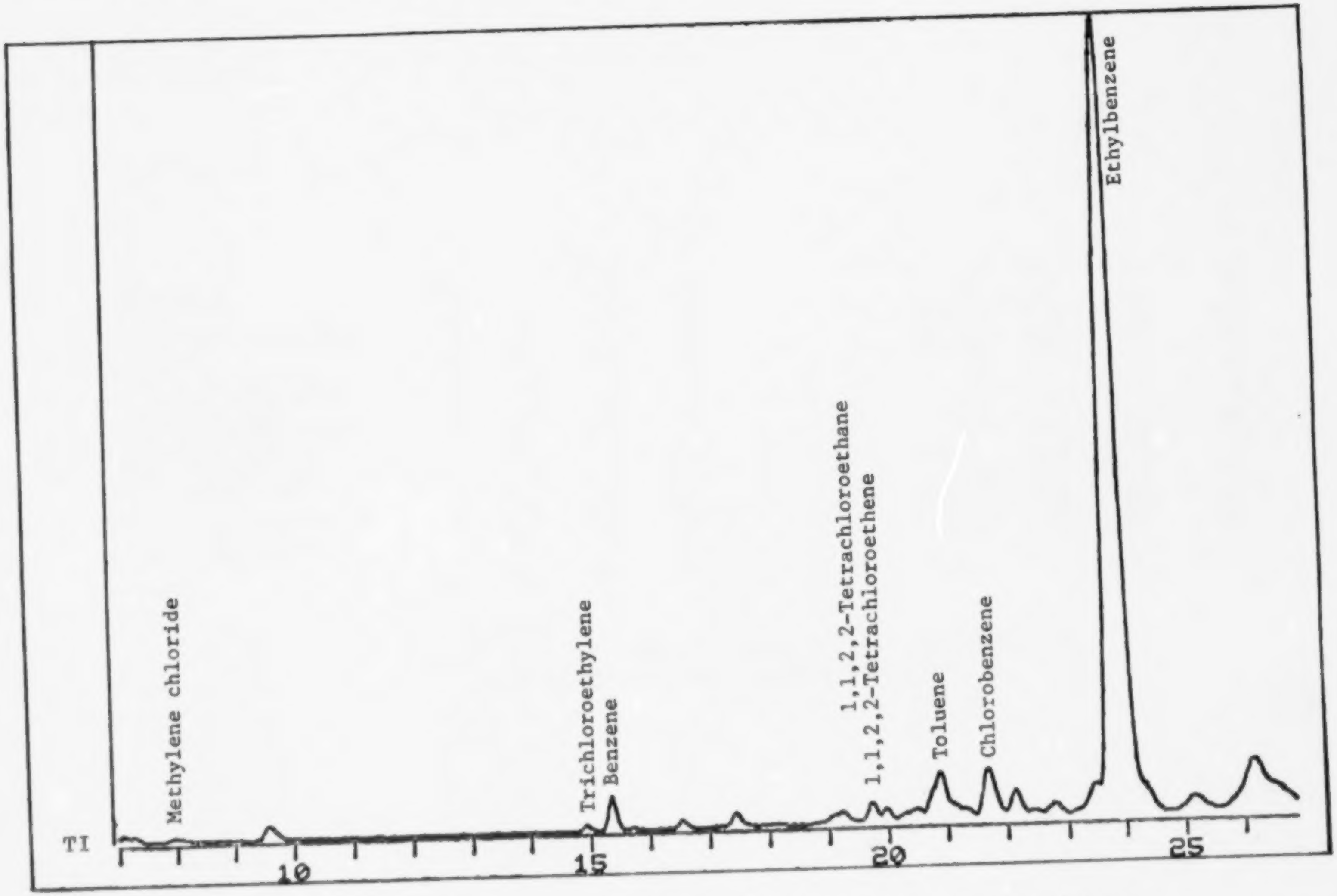


Figure IV-16. GC/MS chromatogram of purgeable organics by purge and trap—
Paint Pigment Sludge, unspiked

## TABLE IV-26. PURGEABLE ORGANICS DATA — PAINT PIGMENT SLUDGE, SPIKE LEVEL I

| COMPOUND | ADDED | NO_1 | NO_2 | NO_3 | NO_4 | NO_5 | MEAN | S | RSD | RECOVERY |
|---|---|---|---|---|---|---|---|---|---|---|
| METHYLENE CHLORIDE | 2 | 2.5 | 7.4 | 4.4 | . | . | 4.76667 | 2.47 | 51.8 | 201 |
| 1,1-DICHLOROETHYLENE | 2 | 2.4 | 2.2 | 2.6 | . | . | 2.4 | 0.20 | 8.3 | 120 |
| 1,1-DICHLOROETHANE | 2 | 2.3 | 2.3 | 2.2 | . | . | 2.26667 | 0.06 | 2.5 | 113 |
| TRANS-1,2-DICHLOROETHYLENE | 2 | 3 | 2.9 | 2.7 | . | . | 2.86667 | 0.15 | 5.3 | 143 |
| CHLOROFORM | 2 | 2.2 | 2.2 | 2.3 | . | . | 2.23333 | 0.06 | 2.6 | 112 |
| 1,2-DICHLOROETHANE | 2 | 4.5 | 5.8 | 4.7 | . | . | 4.96667 | 0.74 | 14.8 | 248 |
| 1,1,1-TRICHLOROETHANE | 2 | 1.5 | 1.5 | 1.7 | . | . | 1.56667 | 0.12 | 7.4 | 78 |
| CARBON TETRACHLORIDE | 2 | 1.3 | 1.4 | 1.6 | . | . | 1.43333 | 0.15 | 10.7 | 72 |
| BROMODICHLOROMETHANE | 2 | 2.6 | 2.5 | 2.7 | . | . | 2.6 | 0.10 | 3.8 | 130 |
| 1,2-DICHLOROPROPANE | 2 | 3.5 | 3.3 | 4 | . | . | 3.6 | 0.36 | 10.0 | 180 |
| TRANS-1,3-DICHLOROPROPENE | 2 | 2.2 | 2.4 | 2.9 | . | . | 2.5 | 0.36 | 14.4 | 125 |
| TRICHLOROETHYLENE | 56 | 0 | 0 | 0 | . | . | 0 | Z | . | N |
| DIBROMOCHLOROMETHANE | 2 | 0 | 0 | 0 | . | . | 0 | Z | . | 0 |
| CIS-1,3-DICHLOROPROPENE | 2 | 0 | 0 | 0 | . | . | 0 | Z | . | 0 |
| 1,1,2-TRICHLOROETHANE | 2 | 0 | 0 | 0 | . | . | 0 | Z | . | 0 |
| BENZENE | 99 | 108 | 120 | 122 | . | . | 116.667 | 7.57 | 6.5 | 114 |
| BROMOFORM | 2 | 1.4 | 1.8 | 1.3 | . | . | 1.5 | 0.26 | 17.6 | 75 |
| 1,1,2,2-TETRACHLOROETHENE | 2 | 10 | 11 | 11 | . | . | 10.6667 | 0.58 | 5.4 | 160 |
| 1,1,2,2-TETRACHLOROETHANE | 14 | 10 | 9.2 | 8.9 | . | . | 9.36667 | 0.57 | 6.1 | 42 |
| TOLUENE | 2 | 0 | 0 | 0 | . | . | 0 | Z | . | N |
| CHLOROBENZENE | 2 | 2.9 | 4.3 | 4.2 | . | . | 3.8 | 0.78 | 20.6 | 87 |
| ETHYLBENZENE | 1600 | 0 | 0 | 0 | . | . | 0 | Z | . | N |

TABLE IV-27. PURGEABLE ORGANICS DATA—
PAINT PIGMENT SLUDGE, SPIKE LEVEL II

| COMPOUND | ADDED | NO_1 | NO_2 | NO_3 | NO_4 | NO_5 | MEAN | S | RSD | RECOVERY |
|---|---|---|---|---|---|---|---|---|---|---|
| METHYLENE CHLORIDE | 9.9 | 19 | 16 | 16 | . | . | 17 | 1.73 | 10.2 | 164 |
| 1,1-DICHLOROETHYLENE | 9.9 | 12 | 11 | 11 | . | . | 11.3333 | 0.58 | 5.1 | 114 |
| 1,1-DICHLOROETHANE | 10 | 12 | 12 | 12 | . | . | 12 | 0.00 | 0.0 | 120 |
| TRANS-1,2-DICHLOROETHYLENE | 10 | 16 | 15 | 14 | . | . | 15 | 1.00 | 6.7 | 150 |
| CHLOROFORM | 10 | 12 | 11 | 12 | . | . | 11.6667 | 0.58 | 4.9 | 117 |
| 1,2-DICHLOROETHANE | 10 | 26 | 27 | 24 | . | . | 25.6667 | 1.53 | 6.0 | 257 |
| 1,1,1-TRICHLOROETHANE | 10 | 9.3 | 8.5 | 8.3 | . | . | 8.7 | 0.53 | 6.1 | 87 |
| CARBON TETRACHLORIDE | 10 | 8.1 | 7.6 | 7 | . | . | 7.56667 | 0.55 | 7.3 | 76 |
| BROMODICHLOROMETHANE | 9.9 | 12 | 15 | 12 | . | . | 13 | 1.73 | 13.3 | 131 |
| 1,2-DICHLOROPROPANE | 10 | 23 | 22 | 20 | . | . | 21.6667 | 1.53 | 7.1 | 217 |
| TRANS-1,3-DICHLOROPROPENE | 10 | 15 | 15 | 14 | . | . | 14.6667 | 0.58 | 3.9 | 147 |
| TRICHLOROETHYLENE | 200 | 0 | 0 | 0 | . | . | 0 | Z | . | N |
| DIBROMOCHLOROMETHANE | 10 | 0 | 0 | 0 | . | . | 0 | Z | . | 0 |
| CIS-1,3-DICHLOROPROPENE | 8.1 | 0 | 0 | 0 | . | . | 0 | Z | . | 0 |
| 1,1,2-TRICHLOROETHANE | 10 | 0 | 0 | 0 | . | . | 0 | Z | . | 0 |
| BENZENE | 494 | 577 | 598 | 706 | . | . | 627 | 69.22 | 11.0 | 126 |
| BROMOFORM | 10 | 11 | 11 | 13 | . | . | 11.6667 | 1.15 | 9.9 | 117 |
| 1,1,2,2-TETRACHLOROETHENE | 10 | 51 | 66 | 74 | . | . | 63.6667 | 11.68 | 18.3 | 562 |
| 1,1,2,2-TETRACHLOROETHANE | 70 | 49 | 62 | 39 | . | . | 50 | 11.53 | 23.1 | 66 |
| TOLUENE | 10 | 22 | 21 | 22 | . | . | 21.6667 | 0.58 | 2.7 | 77 |
| CHLOROBENZENE | 10 | 7.9 | 17 | 14 | . | . | 12.9667 | 4.64 | 35.8 | 109 |
| ETHYLBENZENE | 8000 | 0 | 0 | 0 | . | . | 0 | Z | . | N |

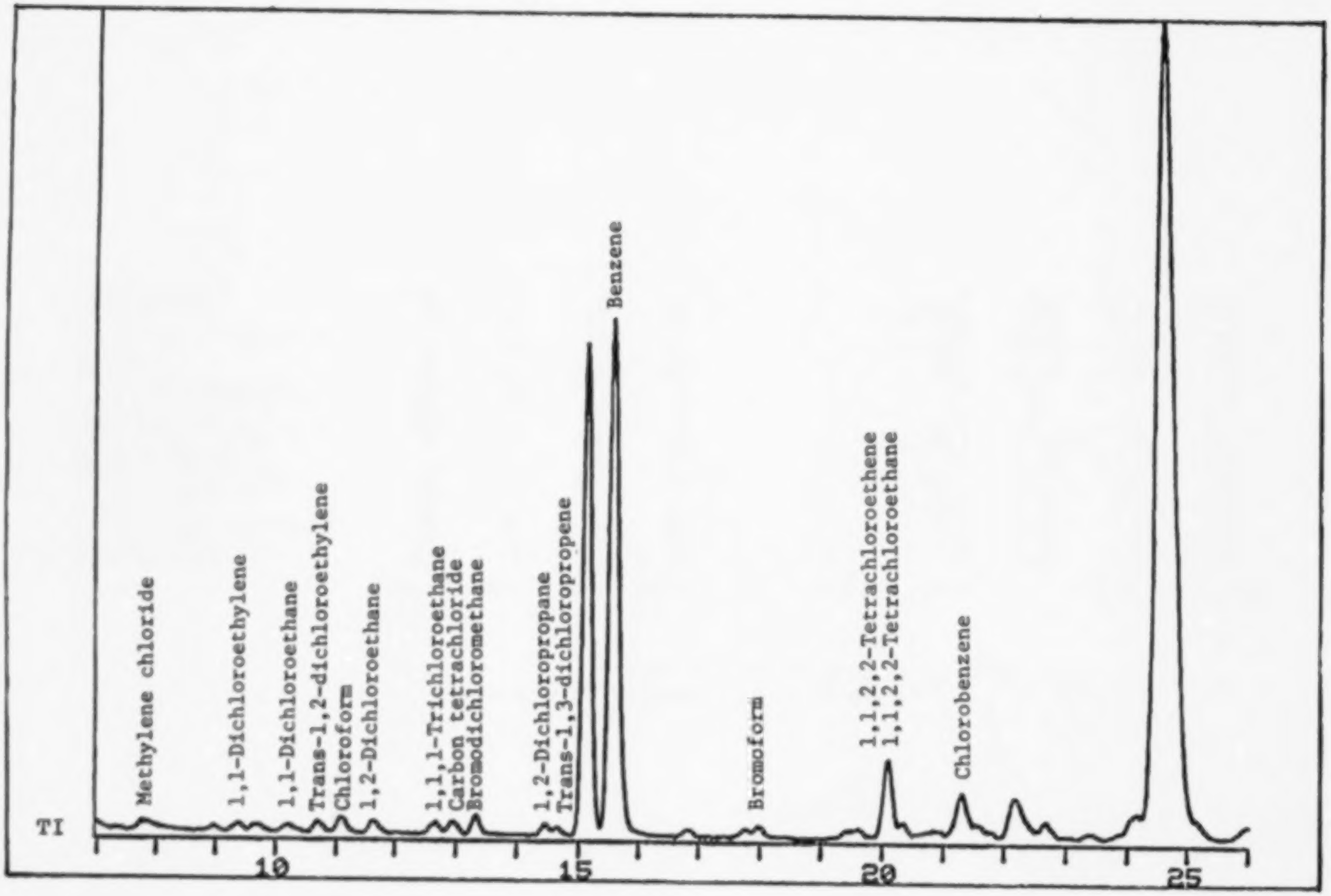


Figure IV-17. GC/MS chromatogram of purgeable organics by purge and trap—Paint Pigment Sludge, spiked

## TABLE IV-28. BASE/NEUTRAL EXTRACTABLE SEMIVOLATILE ORGANICS DATA— PAINT PIGMENT SLUDGE, SPIKE LEVEL 0

| COMPOUND | ADDED | NO_1 | NO_2 | NO_3 | NO_4 | NO_5 | MEAN | S | RSD | RECOVERY |
|---|---|---|---|---|---|---|---|---|---|---|
| 1,3-DICHLOROBENZENE | 0 | 0 | 0 | 0 | . | . | 0 | [illegible] | . | . |
| 1,4-DICHLOROBENZENE | 0 | 0 | 0 | 0 | . | . | 0 | [illegible] | . | . |
| 1,2-DICHLOROBENZENE | 0 | 0 | 0 | 0 | . | . | 0 | [illegible] | . | . |
| HEXACHLOROETHANE | 0 | 0 | 0 | 0 | . | . | 0 | [illegible] | . | . |
| BIS(2-CHLOROETHYL) ETHER | 0 | 0 | 0 | 0 | . | . | 0 | [illegible] | . | . |
| BIS(2-CHLOROISOPROPYL) ETHER | 0 | 0 | 0 | 0 | . | . | 0 | [illegible] | . | . |
| HEXACHLOROBUTADIENE | 0 | 0 | 0 | 0 | . | . | 0 | [illegible] | . | . |
| NITROBENZENE | 0 | 0 | 0 | 0 | . | . | 0 | [illegible] | . | . |
| NAPHTHALENE | 0 | 9. | 7. | 7. | . | . | 8.3333 | 1.01 | 12.1 | . |
| 1,2,4-TRICHLOROBENZENE | 0 | 0 | 0 | 0 | . | . | 0 | [illegible] | . | . |
| BIS(2-CHLOROETHOXY)METHANE | 0 | 0 | 0 | 0 | . | . | 0 | [illegible] | . | . |
| N-NITROSODI-N-PROPYLAMINE | 0 | 0 | 0 | 0 | . | . | 0 | [illegible] | . | . |
| HEXACHLOROCYCLOPENTADIENE | 0 | 0 | 0 | 0 | . | . | 0 | [illegible] | . | . |
| 2-CHLORONAPHTHALENE | 0 | 0 | 0 | 0 | . | . | 0 | [illegible] | . | . |
| ISOPHORONE | 0 | 0 | 0 | 0 | . | . | 0 | [illegible] | . | . |
| ACENAPHTHYLENE | 0 | 0 | 0 | 0 | . | . | 0 | [illegible] | . | . |
| ACENAPHTHENE | 0 | 0 | 0 | 0 | . | . | 0 | [illegible] | . | . |
| DIMETHYL PHTHALATE | 0 | 0 | 0 | 0 | . | . | 0 | [illegible] | . | . |
| 2,6-DINITROTOLUENE | 0 | 0 | 0 | 0 | . | . | 0 | [illegible] | . | . |
| FLUORENE | 0 | 0 | 0 | 0 | . | . | 0 | [illegible] | . | . |
| 2,4-DINITROTOLUENE | 0 | 0 | 0 | 0 | . | . | 0 | [illegible] | . | . |
| 1,2-DIPHENYLHYDRAZINE | 0 | 0 | 0 | 0 | . | . | 0 | [illegible] | . | . |
| 4-CHLOROPHENYL PHENYL ETHER | 0 | 0 | 0 | 0 | . | . | 0 | [illegible] | . | . |
| DIETHYL PHTHALATE | 0 | 1. | < | < | . | . | <1. | 0.17 | 15.7 | . |
| N-NITROSODIPHENYLAMINE | 0 | 0 | 0 | 0 | . | . | 0 | [illegible] | . | . |
| HEXACHLOROBENZENE | 0 | 0 | 0 | 0 | . | . | 0 | [illegible] | . | . |
| 4-BROMOPHENYL PHENYL ETHER | 0 | 0 | 0 | 0 | . | . | 0 | [illegible] | . | . |
| PHENANTHRENE/ANTHRACENE | 0 | 0 | 0 | 0 | . | . | 0 | [illegible] | . | . |
| DI-N-BUTYL PHTHALATE | 0 | 0 | 1. | 1. | . | . | 1.5666 | 0.45 | 28.8 | . |
| FLUORANTHENE | 0 | 0 | 0 | 0 | . | . | 0 | [illegible] | . | . |
| PYRENE | 0 | 0 | 0 | 0 | . | . | 0 | [illegible] | . | . |
| BENZIDINE | 0 | 0 | 0 | 0 | . | . | 0 | [illegible] | . | . |
| BUTYL BENZYL PHTHALATE | 0 | 0 | 0 | 0 | . | . | 0 | [illegible] | . | . |
| BIS(2-ETHYLHEXYL) PHTHALATE | 0 | 0 | 7. | 0 | . | . | 7.2666 | 2.05 | 28.2 | . |
| CHRYSENE/BENZO(A)ANTHRACENE | 0 | 0 | 0 | 0 | . | . | 0 | [illegible] | . | . |
| 3,3'-DICHLOROBENZIDINE | 0 | 0 | 0 | 0 | . | . | 0 | [illegible] | . | . |
| DI-N-OCTYL PHTHALATE | 0 | 0 | 0 | 0 | . | . | 0 | [illegible] | . | . |
| BENZO(B)/BENZO(K)FLUORANTHENES | 0 | 0 | 0 | 0 | . | . | 0 | [illegible] | . | . |
| BENZO(A)PYRENE | 0 | 0 | 0 | 0 | . | . | 0 | [illegible] | . | . |
| INDENO(1,2,3-CD)PYRENE | 0 | 0 | 0 | 0 | . | . | 0 | [illegible] | . | . |
| DIBENZO(A,H)ANTHRACENE | 0 | 0 | 0 | 0 | . | . | 0 | [illegible] | . | . |
| BENZO(GHI)PERYLENE | 0 | 0 | 0 | 0 | . | . | 0 | [illegible] | . | . |

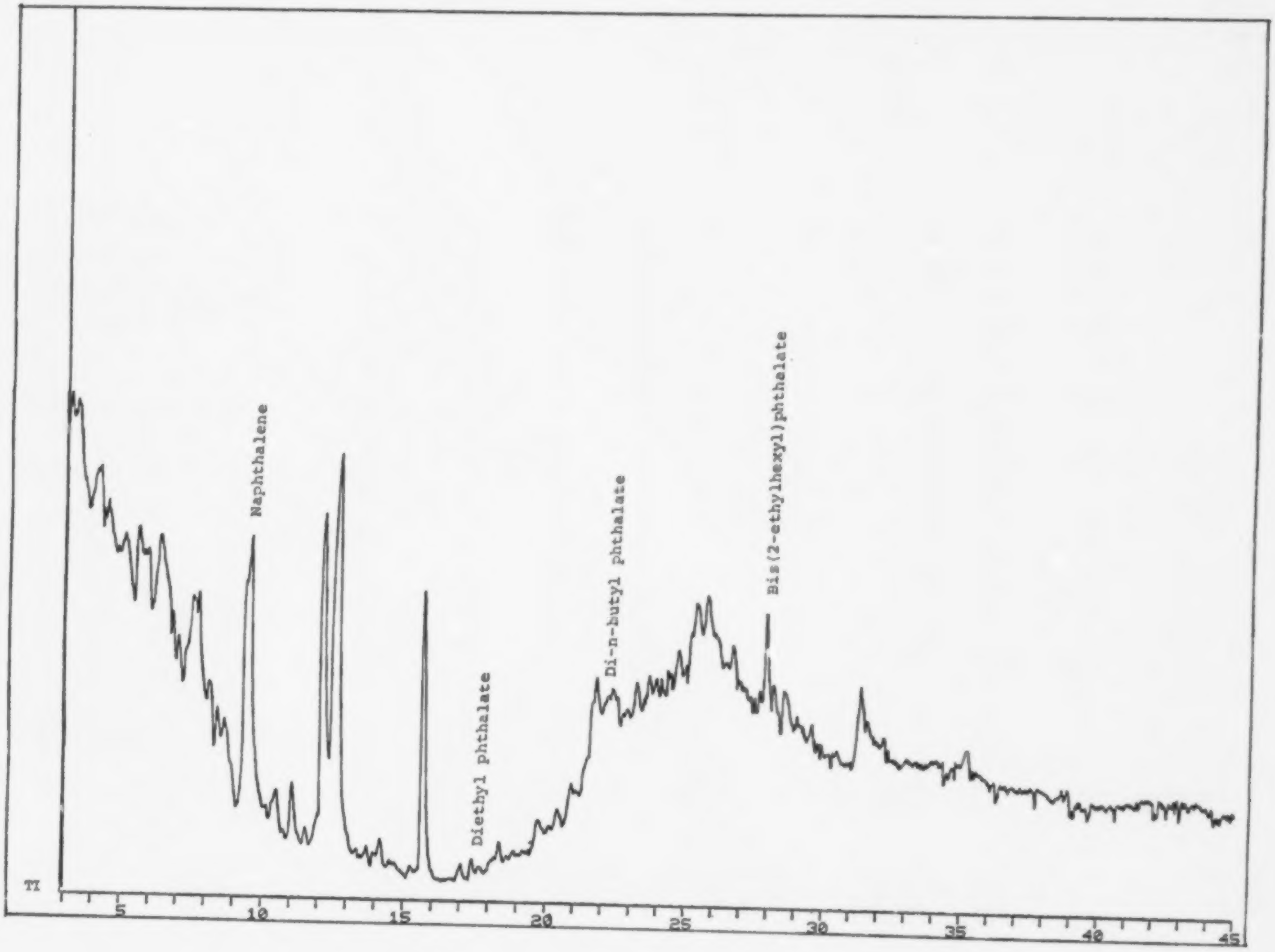


Figure IV-18. GC/MS chromatogram of base/neutral extractables—Paint Pigment Sludge, unspiked

TABLE IV-29. BASE/NEUTRAL EXTRACTABLE SEMIVOLATILE ORGANICS DATA—
PAINT PIGMENT SLUDGE, SPIKE LEVEL I

| COMPOUND | ADDED | NO_1 | NO_2 | NO_3 | NO_4 | NO_5 | MEAN | S | RSD | RECOVERY |
|---|---|---|---|---|---|---|---|---|---|---|
| 1,3-DICHLOROBENZENE | 1.71 | <0.5 | <0.5 | <0.5 | . | . | <0.5 | Z | . | 29 |
| 1,4-DICHLOROBENZENE | 2.19 | <0.5 | <0.5 | <0.5 | . | . | <0.5 | Z | . | 23 |
| 1,2-DICHLOROBENZENE | 2 | <0.5 | <0.5 | <0.5 | . | . | <0.5 | Z | . | 25 |
| HEXACHLOROETHANE | 2 | 2 | 1.4 | 1.2 | . | . | 1.53333 | 0.42 | 27.2 | 77 |
| BIS(2-CHLOROETHYL) ETHER | 2 | 3.3 | 1.7 | 1.2 | . | . | 2.06667 | 1.10 | 53.1 | 103 |
| BIS(2-CHLOROISOPROPYL) ETHER | 2 | <0.5 | <0.5 | <0.5 | . | . | <0.5 | Z | . | 25 |
| HEXACHLOROBUTADIENE | 2.05 | 0.58 | <0.5 | <0.5 | . | . | <.526667 | 0.05 | 8.8 | 26 |
| NITROBENZENE | 2.01 | 3.3 | 2.6 | 3 | . | . | 2.96667 | 0.35 | 11.8 | 148 |
| NAPHTHALENE | 2 | 4.4 | 3.4 | 4.1 | . | . | 3.96667 | 0.51 | 12.9 | N |
| 1,2,4-TRICHLOROBENZENE | 2.2 | 0.9 | 0.59 | 0.51 | . | . | 0.666667 | 0.21 | 30.9 | 30 |
| BIS(2-CHLOROETHOXY)METHANE | 2.02 | 1 | 1.7 | 1.1 | . | . | 1.26667 | 0.38 | 29.9 | 63 |
| N-NITROSODI-N-PROPYLAMINE | 2 | 8.5 | 6.5 | 7.3 | . | . | 7.43333 | 1.01 | 13.5 | 372 |
| HEXACHLOROCYCLOPENTADIENE | 2.02 | <0.5 | <0.5 | <0.5 | . | . | <0.5 | Z | . | 25 |
| 2-CHLORONAPHTHALENE | 1.92 | 1.1 | 1 | 0.82 | . | . | 0.973333 | 0.14 | 14.6 | 51 |
| ISOPHORONE | 2.02 | 0.98 | 1.3 | 1.2 | . | . | 1.16 | 0.16 | 14.1 | 57 |
| ACENAPHTHYLENE | 2.02 | 0.77 | 0.86 | 0.65 | . | . | 0.76 | 0.11 | 13.9 | 38 |
| ACENAPHTHENE | 2.01 | 0.76 | 0.86 | 0.68 | . | . | 0.766667 | 0.09 | 11.8 | 38 |
| DIMETHYL PHTHALATE | 2.01 | 0.61 | 0.85 | 0.7 | . | . | 0.72 | 0.12 | 16.8 | 36 |
| 2,6-DINITROTOLUENE | 2.01 | <0.5 | <0.5 | <0.5 | . | . | <0.5 | Z | . | 25 |
| FLUORENE | 2.01 | 0.74 | 0.71 | 0.71 | . | . | 0.72 | 0.02 | 2.4 | 36 |
| 2,4-DINITROTOLUENE | 2.03 | 0.62 | 0.62 | 0.48 | . | . | 0.573333 | 0.08 | 14.1 | 28 |
| 1,2-DIPHENYLHYDRAZINE | 2 | 0.5 | 0.86 | 0.77 | . | . | 0.71 | 0.19 | 26.4 | 35 |
| 4-CHLOROPHENYL PHENYL ETHER | 2.01 | 0.63 | 0.72 | 0.71 | . | . | 0.686667 | 0.05 | 7.2 | 34 |
| DIETHYL PHTHALATE | 2.02 | 1.1 | 1.7 | 0.97 | . | . | 1.25667 | 0.39 | 31.0 | 8 |
| N-NITROSODIPHENYLAMINE | 2 | <0.5 | <0.5 | <0.5 | . | . | <0.5 | Z | . | 25 |
| HEXACHLOROBENZENE | 1.61 | <0.5 | <0.5 | <0.5 | . | . | <0.5 | Z | . | 31 |
| 4-BROMOPHENYL PHENYL ETHER | 2.07 | 1 | 1.1 | 1.3 | . | . | 1.13333 | 0.15 | 13.5 | 55 |
| PHENANTHRENE/ANTHRACENE | 4.04 | 1.7 | 2.1 | 1.9 | . | . | 1.9 | 0.20 | 10.5 | 47 |
| DI-N-BUTYL PHTHALATE | 2.01 | 1.6 | 2.5 | 1.8 | . | . | 1.96667 | 0.47 | 24.0 | 23 |
| FLUORANTHENE | 2.01 | 0.76 | 1.7 | 1 | . | . | 1.15333 | 0.49 | 42.3 | 57 |
| PYRENE | 2.02 | 1.1 | 1.7 | 1.1 | . | . | 1.3 | 0.35 | 26.6 | 64 |
| BENZIDINE | 2.01 | <0.5 | <0.5 | <0.5 | . | . | <0.5 | Z | . | 25 |
| BUTYLBENZYL PHTHALATE | 2.02 | 0.7 | 1.2 | 0.73 | . | . | 0.876667 | 0.28 | 32.0 | 43 |
| BIS(2-ETHYLHEXYL) PHTHALATE | 2.01 | 2 | 3.1 | 2.2 | . | . | 2.43333 | 0.59 | 24.1 | N |
| CHRYSENE/BENZO(A)ANTHRACENE | 2.02 | 0.54 | 0.91 | 0.59 | . | . | 0.68 | 0.20 | 29.5 | 34 |
| 3,3'-DICHLOROBENZIDINE | 2.01 | 0.93 | 1.7 | 1 | . | . | 1.21 | 0.43 | 35.2 | 60 |
| DI-N-OCTYL PHTHALATE | 2.03 | 1.4 | 2.3 | 1.4 | . | . | 1.7 | 0.52 | 30.6 | 84 |
| BENZO(B)/BENZO(K)FLUORANTHENES | 4.42 | 1.1 | 1.2 | 0.82 | . | . | 1.04 | 0.20 | 18.9 | 24 |
| BENZO(A)PYRENE | 2.08 | 0.55 | 0.6 | <0.5 | . | . | <0.55 | 0.05 | 9.1 | 26 |
| INDENO(1,2,3-CD)PYRENE | 0 | <0.5 | <0.5 | <0.5 | . | . | <0.5 | Z | . | . |
| DIBENZO(AH)ANTHRACENE | 0 | <0.5 | <0.5 | <0.5 | . | . | <0.5 | Z | . | . |
| BENZO(GHI)PERYLENE | 2.02 | <0.5 | <0.5 | <0.5 | . | . | <0.5 | Z | . | 25 |

## TABLE IV-30. BASE/NEUTRAL EXTRACTABLE SEMIVOLATILE ORGANICS DATA— PAINT PIGMENT SLUDGE, SPIKE LEVEL II

| COMPOUND | ADDED | NO_1 | NO_2 | NO_3 | NO_4 | NO_5 | MEAN | S | RSD | RECOVERY |
|---|---|---|---|---|---|---|---|---|---|---|
| 1,3-DICHLOROBENZENE | 8.54 | <1 | <1 | <1 | . | . | <1 | Z | . | 12 |
| 1,4-DICHLOROBENZENE | 10.96 | <1 | <1 | <1 | . | . | <1 | Z | . | 9 |
| 1,2-DICHLOROBENZENE | 10.02 | <1 | <1 | <1 | . | . | <1 | Z | . | 10 |
| HEXACHLOROETHANE | 10.02 | 3.1 | 4.4 | 2.7 | . | . | 3.4 | 0.89 | 26.1 | 34 |
| BIS(2-CHLOROETHYL) ETHER | 10 | 12 | 23 | 17 | . | . | 17.3333 | 5.51 | 31.8 | 173 |
| BIS(2-CHLOROISOPROPYL) ETHER | 10.02 | 1.3 | 1.8 | 1.9 | . | . | 1.66667 | 0.32 | 19.3 | 17 |
| HEXACHLOROBUTADIENE | 10.23 | 6.1 | 6.1 | 6.2 | . | . | 6.13333 | 0.06 | 0.9 | 60 |
| NITROBENZENE | 10.03 | 4.5 | 3.3 | 3.4 | . | . | 3.73333 | 0.67 | 17.8 | 37 |
| NAPHTHALENE | 10.01 | 6.2 | 7.2 | 7 | . | . | 6.8 | 0.53 | 7.8 | N |
| 1,2,4-TRICHLOROBENZENE | 11 | <1 | <1 | <1 | . | . | <1 | Z | . | 9 |
| BIS(2-CHLOROETHOXY)METHANE | 10.09 | 2.3 | 2.2 | 2.8 | . | . | 2.43333 | 0.32 | 13.2 | 24 |
| N-NITROSODI-N-PROPYLAMINE | 10.02 | 3.6 | 5.1 | 4.5 | . | . | 4.4 | 0.75 | 17.2 | 44 |
| HEXACHLOROCYCLOPENTADIENE | 10.12 | <1 | <1 | <1 | . | . | <1 | Z | . | 10 |
| 2-CHLORONAPHTHALENE | 9.6 | 5.2 | 7.3 | 6.4 | . | . | 6.3 | 1.05 | 16.7 | 66 |
| ISOPHORONE | 10.08 | 1.2 | 1.3 | <1 | . | . | <1.16667 | 0.15 | 13.1 | 12 |
| ACENAPHTHYLENE | 10.08 | 5.4 | 7.4 | 6.4 | . | . | 6.4 | 1.00 | 15.6 | 63 |
| ACENAPHTHENE | 10.06 | 5.1 | 5.2 | 6 | . | . | 5.43333 | 0.49 | 9.1 | 54 |
| DIMETHYL PHTHALATE | 10.07 | 5.6 | 5.4 | 5.7 | . | . | 5.56667 | 0.15 | 2.7 | 55 |
| 2,6-DINITROTOLUENE | 10.05 | <1 | <1 | (4.7) | . | . | <1 | Z | . | 10 |
| FLUORENE | 10.04 | 6.7 | 7.2 | 7.2 | . | . | 7.03333 | 0.29 | 4.1 | 70 |
| 2,4-DINITROTOLUENE | 10.16 | <1 | (2.0) | <1 | . | . | <1 | Z | . | 10 |
| 1,2-DIPHENYLHYDRAZINE | 10.02 | 3.4 | 4.4 | 4 | . | . | 3.93333 | 0.50 | 12.8 | 39 |
| 4-CHLOROPHENYL PHENYL ETHER | 10.04 | 5.9 | 6.8 | 6.4 | . | . | 6.36667 | 0.45 | 7.1 | 63 |
| DIETHYL PHTHALATE | 10.11 | 6.4 | 7.6 | 7.1 | . | . | 7.03333 | 0.60 | 8.6 | 59 |
| N-NITROSODIPHENYLAMINE | 10 | 3.4 | 5.8 | 5.3 | . | . | 4.83333 | 1.27 | 26.2 | 48 |
| HEXACHLOROBENZENE | 8.07 | 4.1 | 5.3 | 4.3 | . | . | 4.56667 | 0.64 | 14.1 | 57 |
| 4-BROMOPHENYL PHENYL ETHER | 10.33 | 5 | 6.8 | 6.6 | . | . | 6.13333 | 0.99 | 16.1 | 59 |
| PHENANTHRENE/ANTHRACENE | 20.18 | 5.6 | 6.5 | 5.7 | . | . | 5.93333 | 0.49 | 8.3 | 29 |
| DI-N-BUTYL PHTHALATE | 10.05 | 5.5 | 6.6 | 6 | . | . | 6.03333 | 0.55 | 9.1 | 44 |
| FLUORANTHENE | 10.04 | 4.4 | 5.3 | 4.8 | . | . | 4.83333 | 0.45 | 9.3 | 48 |
| PYRENE | 10.09 | 4.5 | 4.4 | 4.5 | . | . | 4.46667 | 0.06 | 1.3 | 44 |
| BENZIDINE | 10.04 | <1 | <1 | <1 | . | . | <1 | Z | . | 10 |
| BUTYLBENZYL PHTHALATE | 10.09 | 2.1 | 2.4 | 2.1 | . | . | 2.2 | 0.17 | 7.9 | 22 |
| BIS(2-ETHYLHEXYL) PHTHALATE | 10.06 | 3 | 3.3 | 3.2 | . | . | 3.16667 | 0.15 | 4.8 | N |
| CHRYSENE/BENZO(A)ANTHRACENE | 10.08 | 4.5 | 5.4 | 4.5 | . | . | 4.8 | 0.52 | 10.8 | 48 |
| 3,3'-DICHLOROBENZIDINE | 10.04 | 2.7 | 2.8 | 2.2 | . | . | 2.56667 | 0.32 | 12.5 | 26 |
| DI-N-OCTYL PHTHALATE | 10.13 | 1.4 | 1.3 | <1 | . | . | <1.23333 | 0.21 | 16.9 | 12 |
| BENZO(B)/BENZO(K)FLUORANTHENES | 22.11 | 22 | 27 | 22 | . | . | 23.6667 | 2.89 | 12.2 | 107 |
| BENZO(A)PYRENE | 10.4 | 11 | 9.8 | 9.8 | . | . | 10.2 | 0.69 | 6.8 | 98 |
| INDENO(1,2,3-CD)PYRENE | 0 | <1 | <1 | <1 | . | . | <1 | Z | . | . |
| DIBENZO(AH)ANTHRACENE | 0 | <1 | <1 | <1 | . | . | <1 | Z | . | . |
| BENZO(GHI)PERYLENE | 10.12 | 8.6 | 6.2 | 7.4 | . | . | 7.4 | 1.20 | 16.2 | 73 |

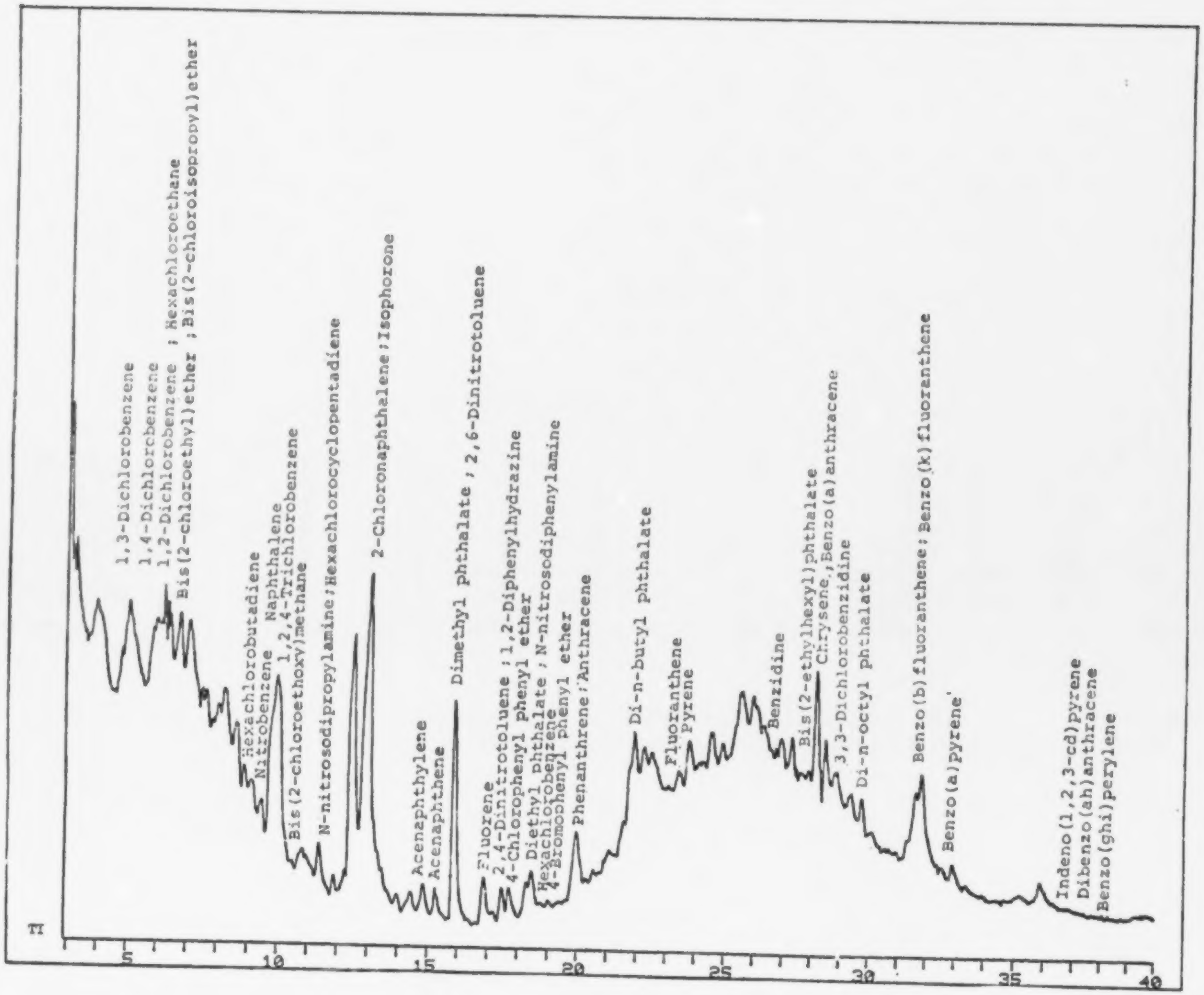


Figure IV-19. GC/MS chromatogram of base/neutral extractables—Paint Pigment Sludge, spiked

TABLE IV-31. ACID EXTRACTABLE SEMIVOLATILE ORGANICS DATA— PAINT PIGMENT SLUDGE, SPIKE LEVEL 0

| COMPOUND | ADDED | NO_1 | NO_2 | NO_3 | NO_4 | NO_5 | MEAN | S | RSD | RECOVERY |
|---|---|---|---|---|---|---|---|---|---|---|
| 2-CHLOROPHENOL | 0 | 2.5 | 1.8 | 0.86 | . | . | 1.72 | 0.82 | 47.8 | . |
| 2-NITROPHENOL | 0 | 0 | 0 | 0 | . | . | 0 | Z | . | . |
| PHENOL | 0 | 0.18 | 0.26 | 0.35 | . | . | 0.263333 | 0.09 | 32.3 | . |
| 2,4-DIMETHYLPHENOL | 0 | 0 | 0 | 0 | . | . | 0 | Z | . | . |
| 2,4-DICHLOROPHENOL | 0 | 0 | 0 | 0 | . | . | 0 | Z | . | . |
| 2,4,6-TRICHLOROPHENOL | 0 | 0 | 0 | 0 | . | . | 0 | Z | . | . |
| 4-CHLORO-3-METHYLPHENOL | 0 | 0 | 0 | 0 | . | . | 0 | Z | . | . |
| 2,4-DINITROPHENOL | 0 | 0 | 0 | 0 | . | . | 0 | Z | . | . |
| 4,6-DINITRO-O-CRESOL | 0 | 0 | 0 | 0 | . | . | 0 | Z | . | . |
| PENTACHLOROPHENOL | 0 | 0 | 0 | 0 | . | . | 0 | Z | . | . |
| 4-NITROPHENOL | 0 | 0 | 0 | 0 | . | . | 0 | Z | . | . |

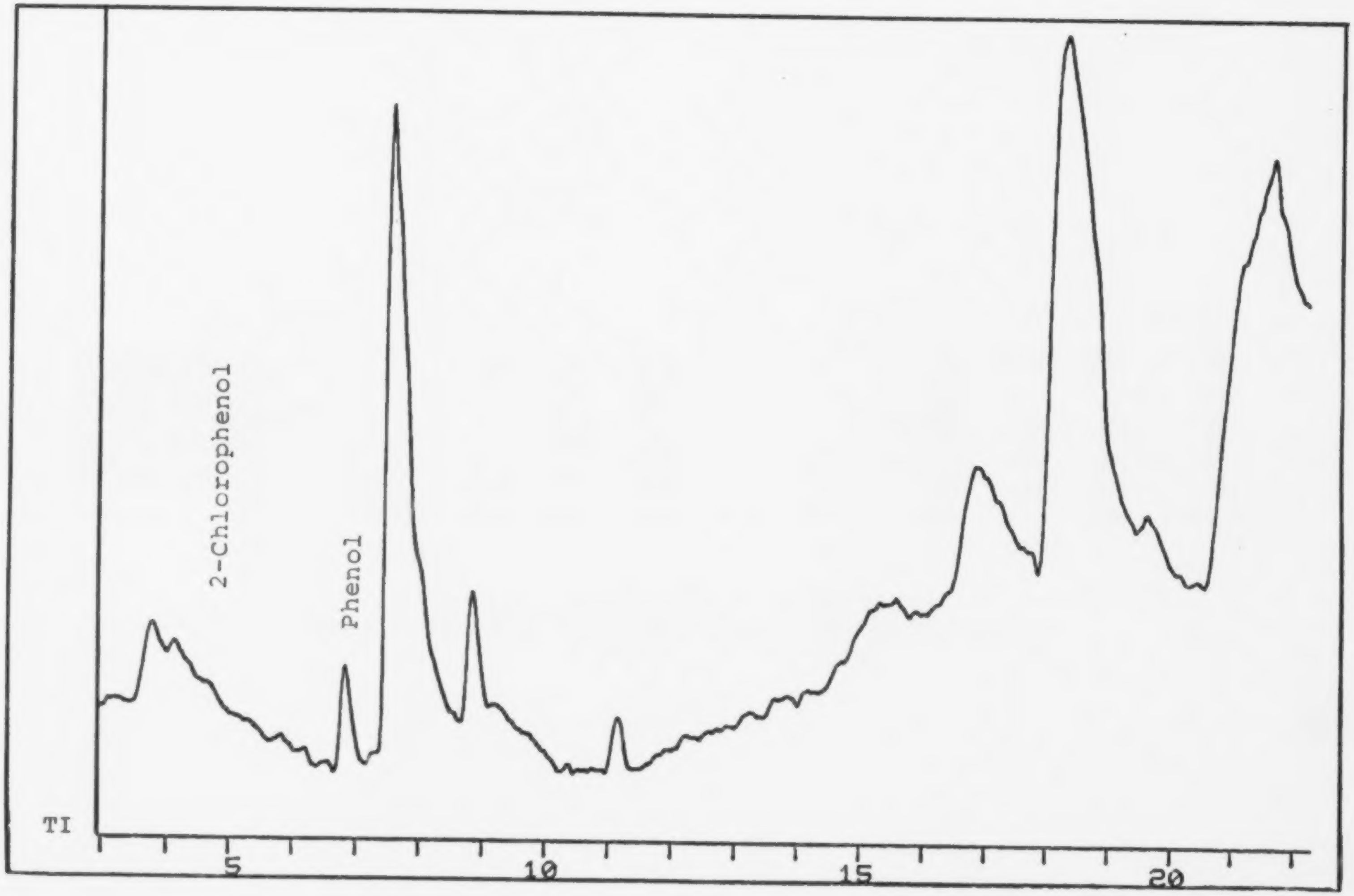


Figure IV-20. GC/MS chromatogram of acid extractables—
Paint Pigment Sludge, unspiked

## TABLE IV-32. ACID EXTRACTABLE SEMIVOLATILE ORGANICS DATA— PAINT PIGMENT SLUDGE, SPIKE LEVEL I

| COMPOUND | ADDED | NO_1 | NO_2 | NO_3 | NO_4 | NO_5 | MEAN | S | RSD | RECOVERY |
|---|---|---|---|---|---|---|---|---|---|---|
| 2-CHLOROPHENOL | 4.01 | 1.3 | 1 | 0.76 | . | . | 1.02 | 0.27 | 26.5 | N |
| 2-NITROPHENOL | 4.02 | <0.5 | <0.5 | <0.5 | . | . | <0.5 | Z | . | 12 |
| PHENOL | 4 | 1.7 | 1 | 0.79 | . | . | 1.16333 | 0.48 | 41.0 | 22 |
| 2,4-DIMETHYLPHENOL | 4.02 | <0.5 | <0.5 | <0.5 | . | . | <0.5 | Z | . | 12 |
| 2,4-DICHLOROPHENOL | 4.04 | 1 | <0.5 | 0.6 | . | . | <0.7 | 0.26 | 37.8 | 17 |
| 2,4,6-TRICHLOROPHENOL | 4.02 | 1.5 | 1.8 | 1.6 | . | . | 1.63333 | 0.15 | 9.4 | 41 |
| 4-CHLORO-3-METHYLPHENOL | 4.01 | <0.5 | <0.5 | <0.5 | . | . | <0.5 | Z | . | 12 |
| 2,4-DINITROPHENOL | 4 | <0.5 | <0.5 | <0.5 | . | . | <0.5 | Z | . | 13 |
| 4,6-DINITRO-O-CRESOL | 4.02 | <0.5 | <0.5 | <0.5 | . | . | <0.5 | Z | . | 12 |
| PENTACHLOROPHENOL | 4.01 | 3.1 | 3.3 | <0.5 | . | . | <2.3 | 1.56 | 67.9 | 57 |
| 4-NITROPHENOL | 4.01 | 1.5 | 1 | <0.5 | . | . | <1 | 0.50 | 50.0 | 25 |

## TABLE IV-33. ACID EXTRACTABLE SEMIVOLATILE ORGANICS DATA— PAINT PIGMENT SLUDGE, SPIKE LEVEL II

| COMPOUND | ADDED | NO_1 | NO_2 | NO_3 | NO_4 | NO_5 | MEAN | S | RSD | RECOVERY |
|---|---|---|---|---|---|---|---|---|---|---|
| 2-CHLOROPHENOL | 10.03 | 8 | 11 | 11 | . | . | 10 | 1.73 | 17.3 | 83 |
| 2-NITROPHENOL | 10.05 | <1 | <1 | <1 | . | . | <1 | Z | . | 10 |
| PHENOL | 10 | 5.6 | 7.3 | 8.3 | . | . | 7.06667 | 1.37 | 19.3 | 68 |
| 2,4-DIMETHYLPHENOL | 10.04 | <1 | <1 | <1 | . | . | <1 | Z | . | 10 |
| 2,4-DICHLOROPHENOL | 10.1 | 7.7 | 6.2 | 10 | . | . | 7.96667 | 1.91 | 24.0 | 79 |
| 2,4,6-TRICHLOROPHENOL | 10.06 | 8 | 6.2 | 10 | . | . | 8.06667 | 1.90 | 23.6 | 80 |
| 4-CHLORO-3-METHYLPHENOL | 10.02 | 2.2 | 3.3 | 2.6 | . | . | 2.7 | 0.56 | 20.6 | 27 |
| 2,4-DINITROPHENOL | 10 | <1 | <1 | <1 | . | . | <1 | Z | . | 10 |
| 4,6-DINITRO-O-CRESOL | 10.04 | <1 | <1 | <1 | . | . | <1 | Z | . | 10 |
| PENTACHLOROPHENOL | 10 | 5 | 7.6 | 6.1 | . | . | 6.23333 | 1.31 | 20.9 | 62 |
| 4-NITROPHENOL | 10.02 | <1 | <1 | <1 | . | . | <1 | Z | . | 10 |

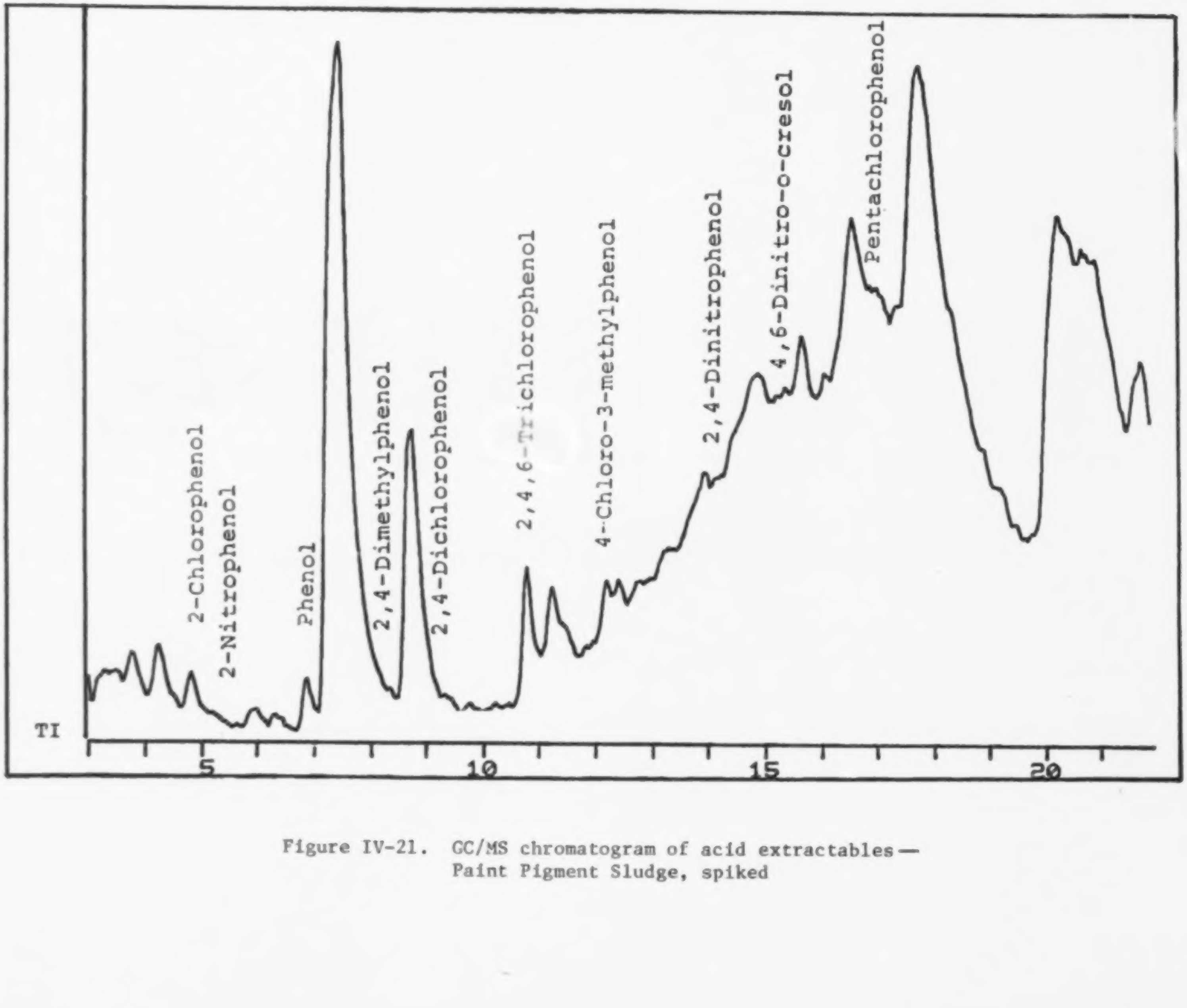


Figure IV-21. GC/MS chromatogram of acid extractables—
Paint Pigment Sludge, spiked

## TABLE IV-34. METALS DATA—
## PAINT PIGMENT SLUDGE, SPIKE LEVEL 0

| ELEMENT | ADDED | NO_1 | NO_2 | NO_3 | NO_4 | NO_5 | MEAN | S | RSD | RECOVERY |
|---|---|---|---|---|---|---|---|---|---|---|
| ANTIMONY | 0 | <0.1 | <0.1 | <0.1 | <0.1 | <0.1 | <0.1 | Z | . | . |
| ARSENIC | 0 | 1.7 | 1.5 | 1.8 | 1.7 | 1.6 | 1.66 | 0.11 | 6.9 | . |
| BERYLLIUM | 0 | <0.1 | <0.1 | <0.1 | <0.1 | <0.1 | <0.1 | Z | . | . |
| CADMIUM | 0 | <0.1 | <0.1 | <0.1 | <0.1 | <0.1 | <0.1 | Z | . | . |
| CHROMIUM | 0 | 14 | 13 | 13 | 14 | 14 | 13.6 | 0.55 | 4.0 | . |
| COPPER | 0 | 184 | 189 | 185 | 185 | 184 | 185.4 | 2.07 | 1.1 | . |
| LEAD | 0 | 47 | 41 | 40 | 49 | 43 | 44 | 3.87 | 8.8 | . |
| MERCURY | 0 | 110 | 103 | 99 | 101 | 103 | 103.2 | 4.15 | 4.0 | . |
| NICKEL | 0 | 1.8 | 2 | 2 | 1.8 | 2.2 | 1.96 | 0.17 | 8.5 | . |
| SELENIUM | 0 | <0.1 | <0.1 | <0.1 | <0.1 | <0.1 | <0.1 | Z | . | . |
| SILVER | 0 | 0.6 | 0.6 | 0.6 | 0.6 | 0.6 | 0.6 | 0.00 | 0.0 | . |
| THALLIUM | 0 | <0.1 | <0.1 | <0.1 | <0.1 | <0.1 | <0.1 | Z | . | . |
| ZINC | 0 | 450 | 468 | 468 | 468 | 468 | 464.4 | 8.05 | 1.7 | . |

## TABLE IV-35. METALS DATA— PAINT PIGMENT SLUDGE, SPIKE LEVEL I

| ELEMENT | ADDED | NO_1 | NO_2 | NO_3 | NO_4 | NO_5 | MEAN | S | RSD | RECOVERY |
|---|---|---|---|---|---|---|---|---|---|---|
| ANTIMONY | 1.8 | <0.2 | <0.2 | <0.2 | <0.2 | <0.2 | <0.2 | Z | . | 6 |
| ARSENIC | 1.8 | 1.7 | 2 | 1.5 | 1.8 | 1.7 | 1.74 | 0.18 | 10.4 | 4 |
| BERYLLIUM | 1.8 | 1.8 | 1.9 | 1.8 | 1.8 | 1.6 | 1.78 | 0.11 | 6.2 | 93 |
| CADMIUM | 1.8 | 1.9 | 1.8 | 2 | 2 | 2.1 | 1.96 | 0.11 | 5.8 | 103 |
| CHROMIUM | 18 | 34 | 34 | 34 | 32 | 30 | 32.8 | 1.79 | 5.5 | 107 |
| COPPER | 180 | 348 | 378 | 376 | 362 | 352 | 363.2 | 13.61 | 3.7 | 99 |
| LEAD | 18 | 63 | 67 | 62 | 58 | 59 | 61.8 | 3.56 | 5.8 | 99 |
| NICKEL | 9 | 8.6 | 8.4 | 7.9 | 8.4 | 7.6 | 8.18 | 0.41 | 5.1 | 69 |
| SELENIUM | 0.9 | <0.1 | <0.1 | <0.1 | <0.1 | <0.1 | <0.1 | Z | . | 0 |
| SILVER | 1.8 | 0.9 | 1 | 0.9 | 1.2 | 1.1 | 1.02 | 0.13 | 12.8 | 23 |
| THALLIUM | 1.8 | 1.5 | 1.6 | 1.3 | 1.3 | 1.3 | 1.4 | 0.14 | 10.1 | 72 |
| ZINC | 360 | 838 | 936 | 921 | 880 | 857 | 886.4 | 41.35 | 4.7 | 117 |

## F. Coke Oven Biological Sludge

## TABLE IV-36. PURGEABLE ORGANICS DATA— COKE OVEN BIOLOGICAL SLUDGE, SPIKE LEVEL 0

| COMPOUND | ADDED | NO_1 | NO_2 | NO_3 | NO_4 | NO_5 | MEAN | S | RSD | RECOVERY |
|---|---|---|---|---|---|---|---|---|---|---|
| METHYLENE CHLORIDE | 0 | 0.12 | 0.055 | 0.087 | . | . | .0873333 | 0.03 | 37.2 | . |
| 1,1-DICHLOROETHYLENE | 0 | 0 | 0 | 0 | . | . | 0 | 0 | . | . |
| 1,1-DICHLOROETHANE | 0 | 0 | 0 | 0 | . | . | 0 | 0 | . | . |
| TRANS-1,2-DICHLOROETHYLENE | 0 | 0 | 0 | 0 | . | . | 0 | 0 | . | . |
| CHLOROFORM | 0 | 0 | 0 | 0 | . | . | 0 | 0 | . | . |
| 1,2-DICHLOROETHANE | 0 | 0 | 0 | 0 | . | . | 0 | 0 | . | . |
| 1,1,1-TRICHLOROETHANE | 0 | 0 | 0 | 0 | . | . | 0 | 0 | . | . |
| CARBON TETRACHLORIDE | 0 | 0 | 0 | 0 | . | . | 0 | 0 | . | . |
| BROMODICHLOROMETHANE | 0 | 0 | 0 | 0 | . | . | 0 | 0 | . | . |
| 1,2-DICHLOROPROPANE | 0 | 0 | 0 | 0 | . | . | 0 | 0 | . | . |
| TRANS-1,3-DICHLOROPROPENE | 0 | 0 | 0 | 0 | . | . | 0 | 0 | . | . |
| TRICHLOROETHYLENE | 0 | 0 | 0 | 0 | . | . | 0 | 0 | . | . |
| DIBROMOCHLOROMETHANE | 0 | 0 | 0 | 0 | . | . | 0 | 0 | . | . |
| CIS-1,3-DICHLOROPROPENE | 0 | 0 | 0 | 0 | . | . | 0 | 0 | . | . |
| 1,1,2-TRICHLOROETHANE | 0 | 0 | 0 | 0 | . | . | 0 | 0 | . | . |
| BENZENE | 0 | 0 | 0 | 0 | . | . | 0 | 0 | . | . |
| BROMOFORM | 0 | 0 | 0 | 0 | . | . | 0 | 0 | . | . |
| 1,1,2,2-TETRACHLOROETHENE | 0 | 0 | 0 | 0 | . | . | 0 | 0 | . | . |
| 1,1,2,2-TETRACHLOROETHANE | 0 | 0 | 0 | 0 | . | . | 0 | 0 | . | . |
| TOLUENE | 0 | 0 | 0 | 0 | . | . | 0 | 0 | . | . |
| CHLOROBENZENE | 0 | 0 | 0 | 0 | . | . | 0 | 0 | . | . |
| ETHYL BENZENE | 0 | 0 | 0 | 0 | . | . | 0 | 0 | . | . |

# TABLE IV-37. PURGEABLE ORGANICS DATA— COKE OVEN BIOLOGICAL SLUDGE, SPIKE LEVEL I

| COMPOUND | ADDED | NO_1 | NO_2 | NO_3 | NO_4 | NO_5 | MEAN | S | RSD | RECOVERY |
|---|---|---|---|---|---|---|---|---|---|---|
| METHYLENE CHLORIDE | 2 | [illegible] | [illegible] | [illegible] | . | . | 1.83333 | 0.29 | 15.7 | [illegible] |
| 1,1-DICHLOROETHYLENE | 2 | [illegible] | [illegible] | [illegible] | . | . | 0.766667 | [illegible] | [illegible] | [illegible] |
| 1,1-DICHLOROETHANE | 2 | [illegible] | [illegible] | [illegible] | . | . | [illegible] | [illegible] | [illegible] | [illegible] |
| TRANS-1,2-DICHLOROETHYLENE | 2 | [illegible] | [illegible] | [illegible] | . | . | [illegible] | [illegible] | [illegible] | [illegible] |
| CHLOROFORM | 2 | [illegible] | [illegible] | [illegible] | . | . | [illegible] | [illegible] | [illegible] | [illegible] |
| 1,2-DICHLOROETHANE | 2 | [illegible] | [illegible] | [illegible] | . | . | [illegible] | [illegible] | [illegible] | [illegible] |
| 1,1,1-TRICHLOROETHANE | 2 | [illegible] | [illegible] | [illegible] | . | . | [illegible] | [illegible] | [illegible] | [illegible] |
| CARBON TETRACHLORIDE | 2 | [illegible] | [illegible] | [illegible] | . | . | [illegible] | [illegible] | [illegible] | [illegible] |
| BROMODICHLOROMETHANE | 2 | [illegible] | [illegible] | [illegible] | . | . | [illegible] | [illegible] | [illegible] | [illegible] |
| 1,2-DICHLOROPROPANE | 2 | [illegible] | [illegible] | [illegible] | . | . | [illegible] | [illegible] | [illegible] | [illegible] |
| TRANS-1,3-DICHLOROPROPENE | 2 | [illegible] | [illegible] | [illegible] | . | . | [illegible] | [illegible] | [illegible] | [illegible] |
| TRICHLOROETHYLENE | 2 | [illegible] | [illegible] | [illegible] | . | . | [illegible] | [illegible] | [illegible] | [illegible] |
| DIBROMOCHLOROMETHANE | 2 | [illegible] | [illegible] | [illegible] | . | . | [illegible] | [illegible] | [illegible] | [illegible] |
| CIS-1,3-DICHLOROPROPENE | 1.6 | [illegible] | [illegible] | [illegible] | . | . | [illegible] | [illegible] | [illegible] | [illegible] |
| 1,1,2-TRICHLOROETHANE | 2 | [illegible] | [illegible] | [illegible] | . | . | [illegible] | [illegible] | [illegible] | [illegible] |
| BENZENE | 2 | [illegible] | [illegible] | [illegible] | . | . | [illegible] | [illegible] | [illegible] | [illegible] |
| BROMOFORM | 2 | [illegible] | [illegible] | [illegible] | . | . | [illegible] | [illegible] | [illegible] | [illegible] |
| 1,1,2,2-TETRACHLOROETHENE | 2 | [illegible] | [illegible] | [illegible] | . | . | [illegible] | [illegible] | [illegible] | [illegible] |
| 1,1,2,2-TETRACHLOROETHANE | 2 | [illegible] | [illegible] | [illegible] | . | . | [illegible] | [illegible] | [illegible] | [illegible] |
| TOLUENE | 2 | [illegible] | [illegible] | [illegible] | . | . | [illegible] | [illegible] | [illegible] | [illegible] |
| CHLOROBENZENE | 2 | [illegible] | [illegible] | [illegible] | . | . | [illegible] | [illegible] | [illegible] | [illegible] |
| ETHYLBENZENE | 2 | [illegible] | [illegible] | [illegible] | . | . | [illegible] | [illegible] | [illegible] | [illegible] |

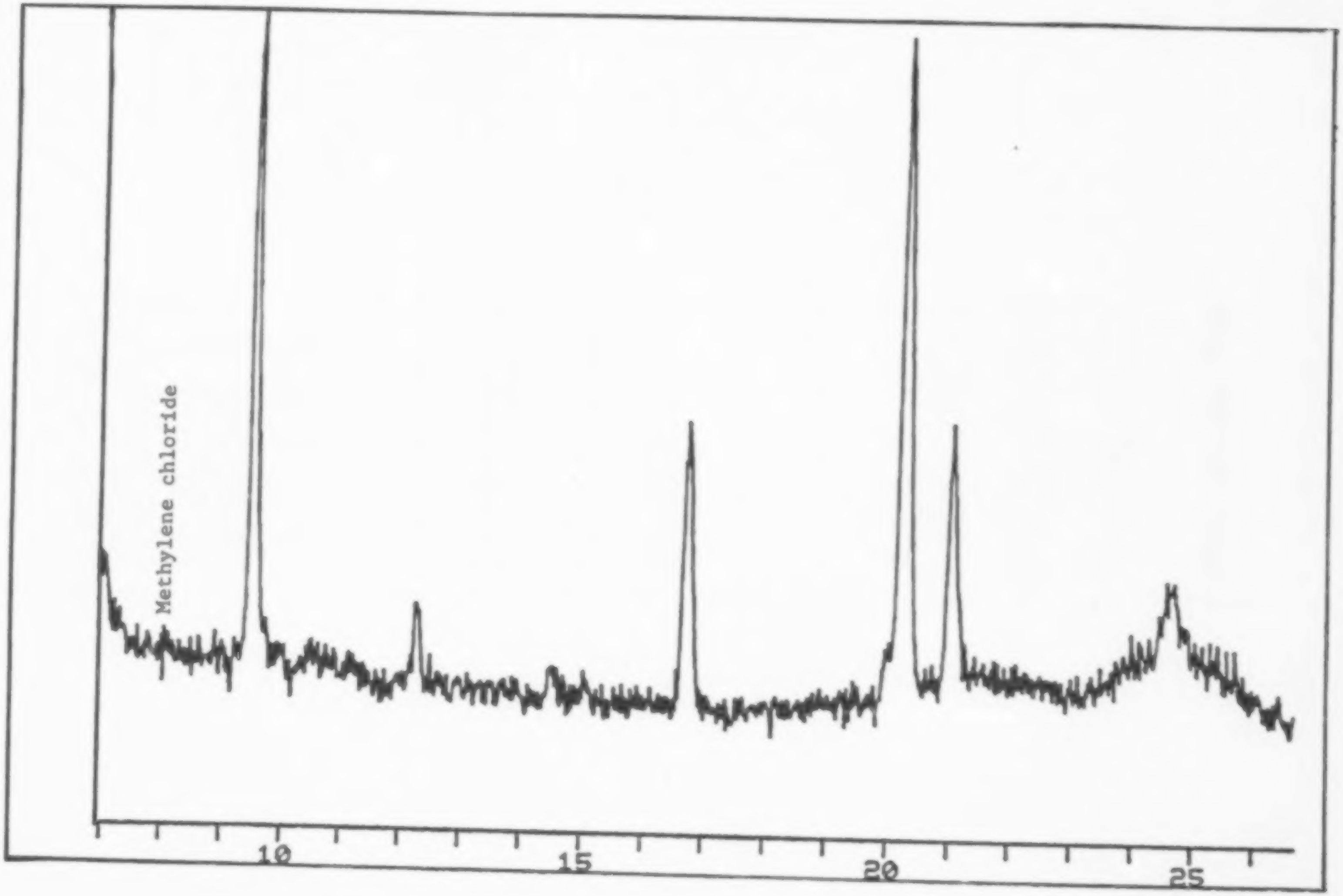


Figure IV-22. GC/MS chromatogram of purgeable organics by purge and trap—Coke Oven Biological Sludge, unspiked

TABLE IV-38. PURGEABLE ORGANICS DATA—
COKE OVEN BIOLOGICAL SLUDGE, SPIKE LEVEL II

| COMPOUND | ADDED | NO_1 | NO_2 | NO_3 | NO_4 | NO_5 | MEAN | S | RSD | RECOVERY |
|---|---|---|---|---|---|---|---|---|---|---|
| METHYLENE CHLORIDE | 9.9 | 9.2 | 11 | 7.9 | . | . | 9.36667 | 1.56 | 16.6 | 94 |
| 1,1-DICHLOROETHYLENE | 9.9 | 4.4 | 4.9 | 4.2 | . | . | 4.5 | 0.36 | 8.0 | 45 |
| 1,1-DICHLOROETHANE | 10 | 8.4 | 9.9 | 7.4 | . | . | 8.56667 | 1.26 | 14.7 | 86 |
| TRANS-1,2-DICHLOROETHYLENE | 10 | 7.7 | 8.4 | 6.6 | . | . | 7.56667 | 0.91 | 12.0 | 76 |
| CHLOROFORM | 10 | 8.5 | 9.9 | 7.9 | . | . | 8.76667 | 1.03 | 11.7 | 88 |
| 1,2-DICHLOROETHANE | 10 | 11 | 12 | 9.5 | . | . | 10.8333 | 1.26 | 11.6 | 108 |
| 1,1,1-TRICHLOROETHANE | 10 | 5.2 | 6.6 | 5.3 | . | . | 5.7 | 0.78 | 13.7 | 57 |
| CARBON TETRACHLORIDE | 10 | 4.5 | 5 | 4.4 | . | . | 4.63333 | 0.32 | 6.9 | 46 |
| BROMODICHLOROMETHANE | 9.9 | 10 | 11 | 9.6 | . | . | 10.2 | 0.72 | 7.1 | 103 |
| 1,2-DICHLOROPROPANE | 10 | 6.8 | 9 | 7.8 | . | . | 7.86667 | 1.10 | 14.0 | 79 |
| TRANS-1,3-DICHLOROPROPENE | 10 | 9.3 | 11 | 8.7 | . | . | 9.66667 | 1.19 | 12.3 | 97 |
| TRICHLORCETHYLENE | 9.9 | 7.9 | 9.4 | 7.3 | . | . | 8.2 | 1.08 | 13.2 | 83 |
| DIBROMOCHLOROMETHANE | 10 | 9.9 | 13 | 9.9 | . | . | 10.9333 | 1.79 | 16.4 | 109 |
| CIS-1,3-DICHLOROPROPENE | 8.1 | 7.8 | 10 | 7.8 | . | . | 8.53333 | 1.27 | 14.9 | 105 |
| 1,1,2-TRICHLOROETHANE | 10 | 11 | 12 | 9.9 | . | . | 10.9667 | 1.05 | 9.6 | 110 |
| BENZENE | 10 | 7.6 | 9 | 7.1 | . | . | 7.9 | 0.98 | 12.5 | 79 |
| BROMOFORM | 10 | 11 | 13 | 10 | . | . | 11.3333 | 1.53 | 13.5 | 113 |
| 1,1,2,2-TETRACHLOROETHENE | 10 | 7.7 | 6.5 | 7.7 | . | . | 7.3 | 0.69 | 9.5 | 73 |
| 1,1,2,2-TETRACHLOROETHANE | 10 | 8.9 | 10 | 8.3 | . | . | 9.06667 | 0.86 | 9.5 | 91 |
| TOLUENE | 10 | 8.6 | 9.4 | 7.4 | . | . | 8.46667 | 1.01 | 11.9 | 85 |
| CHLOROBENZENE | 10 | 9.3 | 11 | 8.2 | . | . | 9.5 | 1.41 | 14.8 | 95 |
| ETHYLBENZENE | 10 | 9 | 9.8 | 8.3 | . | . | 9.03333 | 0.75 | 8.3 | 90 |

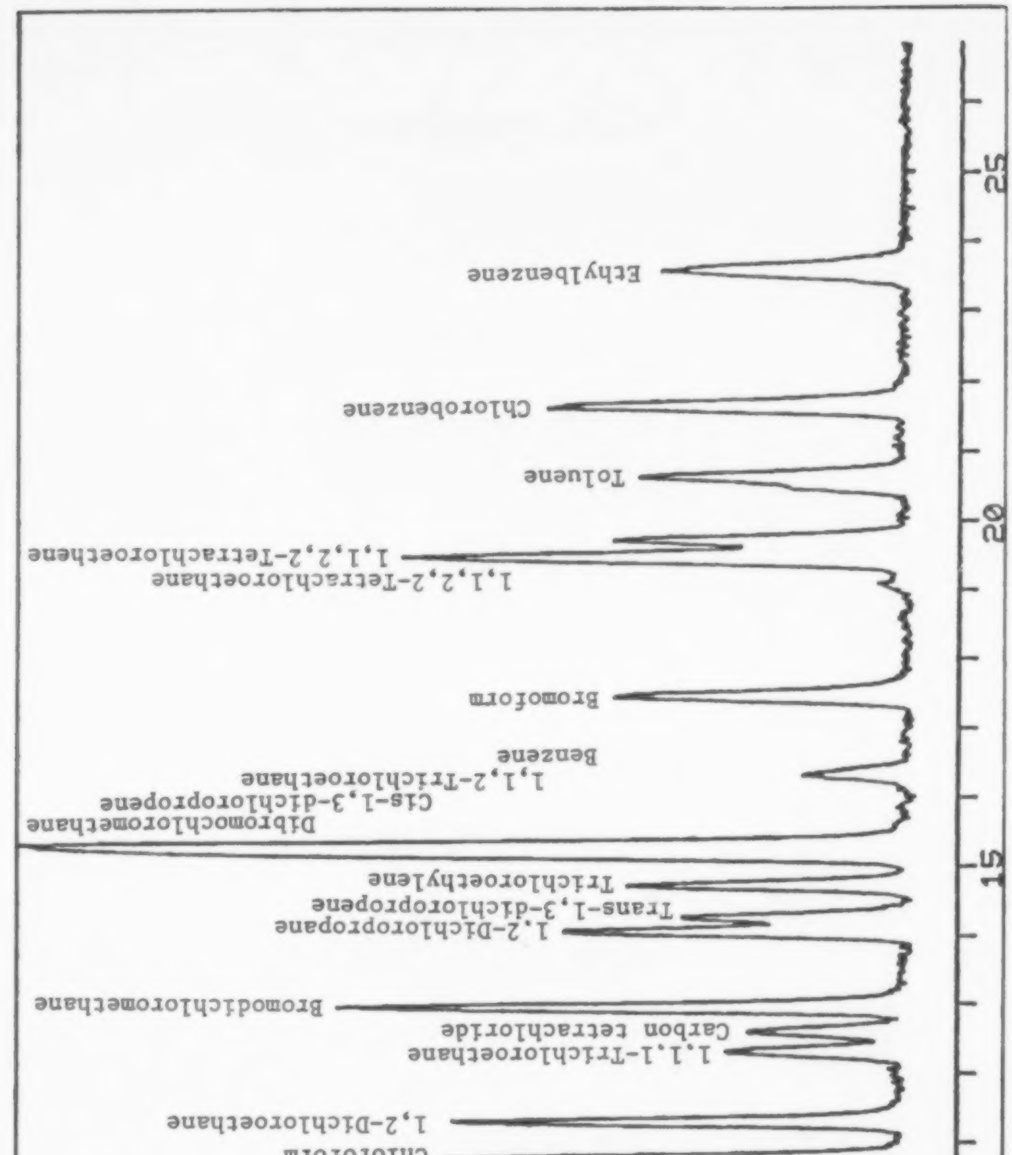


Figure IV-23. GC/MS chromatogram of purgeable organics by purge and trap—Coke Oven Biological Sludge, spiked

## TABLE IV-39. BASE/NEUTRAL EXTRACTABLE SEMIVOLATILE ORGANICS DATA— COKE OVEN BIOLOGICAL SLUDGE, SPIKE LEVEL 0

| COMPOUND | ADDED | NO_1 | NO_2 | NO_3 | NO_4 | NO_5 | MEAN | S | RSD | RECOVERY |
|---|---|---|---|---|---|---|---|---|---|---|
| 1,3-DICHLOROBENZENE | 0 | 0 | 0 | 0 | . | . | 0 | Z | . | . |
| 1,4-DICHLOROBENZENE | 0 | 0 | 0 | 0 | . | . | 0 | Z | . | . |
| 1,2-DICHLOROBENZENE | 0 | 0 | 0 | 0 | . | . | 0 | Z | . | . |
| HEXACHLOROETHANE | 0 | 0 | 0 | 0 | . | . | 0 | Z | . | . |
| BIS(2-CHLOROETHYL) ETHER | 0 | 0 | 0 | 0 | . | . | 0 | Z | . | . |
| BIS(2-CHLOROISOPROPYL) ETHER | 0 | 0 | 0 | 0 | . | . | 0 | Z | . | . |
| HEXACHLOROBUTADIENE | 0 | 0 | 0 | 0 | . | . | 0 | Z | . | . |
| NITROBENZENE | 0 | 0 | 0 | 0 | . | . | 0 | Z | . | . |
| NAPHTHALENE | 0 | 0 | 0 | 0 | . | . | 0 | Z | . | . |
| 1,2,4-TRICHLOROBENZENE | 0 | 0 | 0 | 0 | . | . | 0 | Z | . | . |
| BIS(2-CHLOROETHOXY)METHANE | 0 | 0 | 0 | 0 | . | . | 0 | Z | . | . |
| N-NITROSODI-N-PROPYLAMINE | 0 | 0 | 0 | 0 | . | . | 0 | Z | . | . |
| HEXACHLOROCYCLOPENTADIENE | 0 | 0 | 0 | 0 | . | . | 0 | Z | . | . |
| 2-CHLORONAPHTHALENE | 0 | 0 | 0 | 0 | . | . | 0 | Z | . | . |
| ISOPHORONE | 0 | 0 | 0 | 0 | . | . | 0 | Z | . | . |
| ACENAPHTHYLENE | 0 | 0 | 0 | 0 | . | . | 0 | Z | . | . |
| ACENAPHTHENE | 0 | 0 | 0 | 0 | . | . | 0 | Z | . | . |
| DIMETHYL PHTHALATE | 0 | 0 | 0 | 0 | . | . | 0 | Z | . | . |
| 2,6-DINITROTOLUENE | 0 | 0 | 0 | 0 | . | . | 0 | Z | . | . |
| FLUORENE | 0 | 0 | 0 | 0 | . | . | 0 | Z | . | . |
| 2,4-DINITROTOLUENE | 0 | 0 | 0 | 0 | . | . | 0 | Z | . | . |
| 1,2-DIPHENYLHYDRAZINE | 0 | 0 | 0 | 0 | . | . | 0 | Z | . | . |
| 4-CHLOROPHENYL PHENYL ETHER | 0 | 0 | 0 | 0 | . | . | 0 | Z | . | . |
| DIETHYL PHTHALATE | 0 | 0 | 0 | 0 | . | . | 0 | Z | . | . |
| N-NITROSODIPHENYLAMINE | 0 | 0 | 0 | 0 | . | . | 0 | Z | . | . |
| HEXACHLOROBENZENE | 0 | 0 | 0 | 0 | . | . | 0 | Z | . | . |
| 4-BROMOPHENYL PHENYL ETHER | 0 | 0 | 0 | 0 | . | . | 0 | Z | . | . |
| PHENANTHRENE/ANTHRACENE | 0 | 0 | 0 | 0 | . | . | 0 | Z | . | . |
| DI-N-BUTYL PHTHALATE | 0 | 0 | 0 | 0 | . | . | 0 | Z | . | . |
| FLUORANTHENE | 0 | 0 | 0 | 0 | . | . | 0 | Z | . | . |
| PYRENE | 0 | 0 | 0 | 0 | . | . | 0 | Z | . | . |
| BENZIDINE | 0 | 0 | 0 | 0 | . | . | 0 | Z | . | . |
| BUTYLBENZYL PHTHALATE | 0 | 0 | 0 | 0 | . | . | 0 | Z | . | . |
| BIS(2-ETHYLHEXYL) PHTHALATE | 0 | 0.96 | 1.7 | 1.8 | . | . | 1.48667 | 0.46 | 30.9 | . |
| CHRYSENE/BENZO(A)ANTHRACENE | 0 | <0.5 | <0.5 | <0.5 | . | . | <0.5 | Z | . | . |
| 3,3'-DICHLOROBENZIDINE | 0 | 0 | 0 | 0 | . | . | 0 | Z | . | . |
| DI-N-OCTYL PHTHALATE | 0 | 0 | 0 | 0 | . | . | 0 | Z | . | . |
| BENZO(B)/BENZO(K)FLUORANTHENES | 0 | 0.81 | 0.63 | 0.62 | . | . | 0.686667 | 0.11 | 15.6 | . |
| BENZO(A)PYRENE | 0 | 0.87 | 1.1 | 1.4 | . | . | 1.12333 | 0.27 | 23.7 | . |
| INDENO(1,2,3-CD)PYRENE | 0 | 0 | 0 | (<0.5) | . | . | 0 | Z | . | . |
| DIBENZO(AH)ANTHRACENE | 0 | 0 | 0 | 0 | . | . | 0 | Z | . | . |
| BENZO(GHI)PERYLENE | 0 | 0 | 0 | (<0.5) | . | . | 0 | Z | . | . |

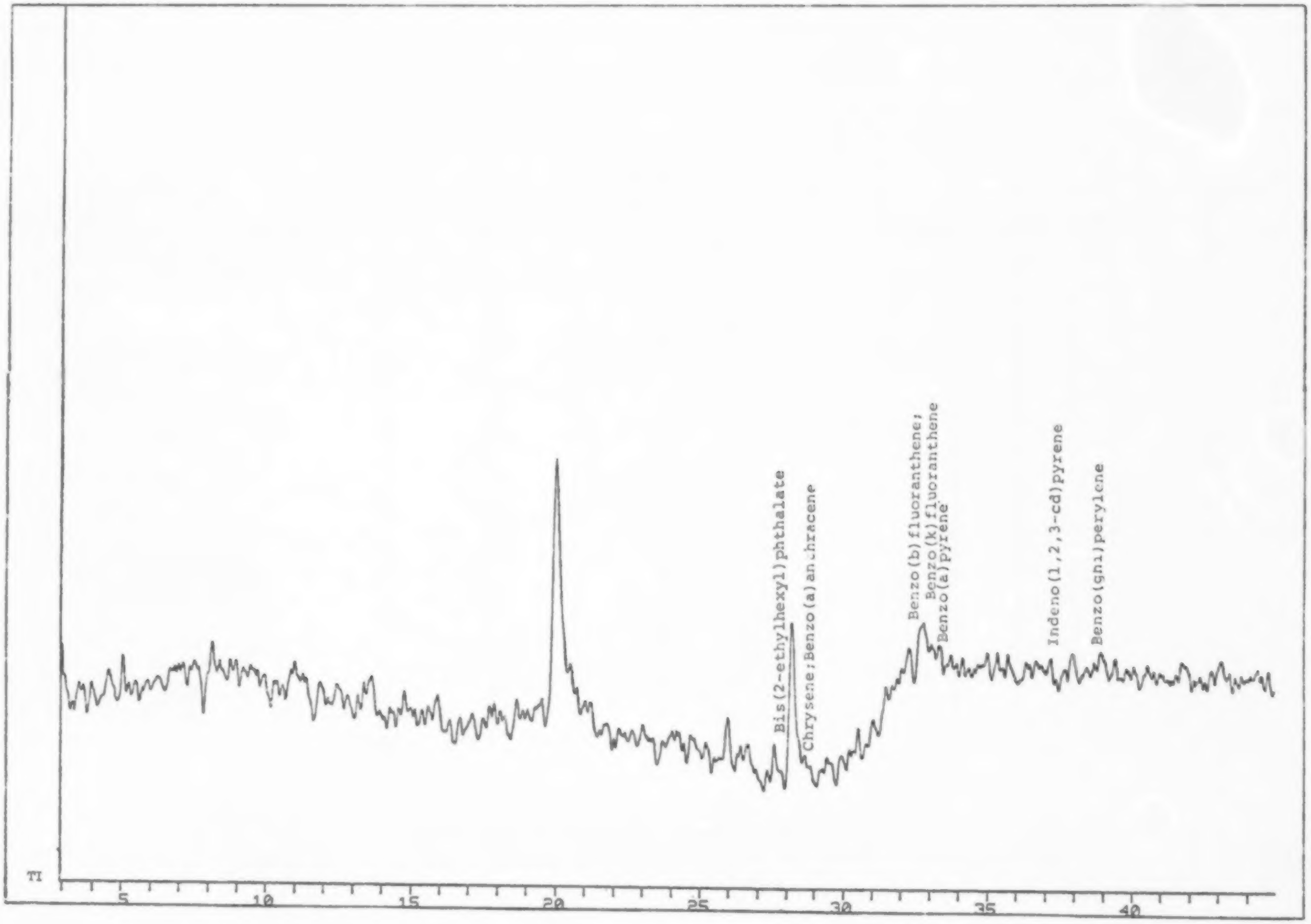


Figure IV-24. GC/MS chromatogram of base/neutral extractables — Coke Oven Biological Sludge, unspiked

## TABLE IV-40. BASE/NEUTRAL EXTRACTABLE SEMIVOLATILE ORGANICS DATA— COKE OVEN BIOLOGICAL SLUDGE, SPIKE LEVEL I

| COMPOUND | ADDED | NO_1 | NO_2 | NO_3 | NO_4 | NO_5 | MEAN | S | RSD | RECOVERY |
|---|---|---|---|---|---|---|---|---|---|---|
| 1,3-DICHLOROBENZENE | 1.71 | <0.5 | <0.5 | 0.52 | . | . | <.506667 | 0.01 | 2.3 | 30 |
| 1,4-DICHLOROBENZENE | 2.19 | 0.82 | 0.52 | 0.8 | . | . | 0.713333 | 0.17 | 23.5 | 33 |
| 1,2-DICHLOROBENZENE | 2 | 1.1 | 1 | 1.1 | . | . | 1.06667 | 0.06 | 5.4 | 53 |
| HEXACHLOROETHANE | 2 | 0.98 | 0.99 | 1.2 | . | . | 1.05667 | 0.12 | 11.8 | 53 |
| BIS(2-CHLOROETHYL) ETHER | 2 | 1.5 | 1.5 | (2.2) | . | . | 1.5 | 0.00 | 0.0 | 75 |
| BIS(2-CHLOROISOPROPYL) ETHER | 2 | 1.8 | 1.8 | 2.2 | . | . | 1.93333 | 0.23 | 11.9 | 97 |
| HEXACHLOROBUTADIENE | 2.05 | 1 | 0.75 | 0.63 | . | . | 0.793333 | 0.19 | 23.8 | 39 |
| NITROBENZENE | 2.01 | 1.9 | 1.7 | 2.9 | . | . | 2.16667 | 0.64 | 29.7 | 108 |
| NAPHTHALENE | 2 | 1.3 | 1.3 | 1.6 | . | . | 1.4 | 0.17 | 12.4 | 70 |
| 1,2,4-TRICHLOROBENZENE | 2.2 | 0.75 | 0.73 | 0.89 | . | . | 0.79 | 0.09 | 11.0 | 36 |
| BIS(2-CHLOROETHOXY)METHANE | 2.02 | 2.2 | 2 | 2.6 | . | . | 2.26667 | 0.31 | 13.5 | 112 |
| N-NITROSODI-N-PROPYLAMINE | 2 | 2.6 | 0.84 | 1.6 | . | . | 1.68 | 0.88 | 52.5 | 84 |
| HEXACHLOROCYCLOPENTADIENE | 2.02 | <0.5 | <0.5 | <0.5 | . | . | <0.5 | . | . | 25 |
| 2-CHLORONAPHTHALENE | 1.92 | 1.4 | 1.4 | 2 | . | . | 1.6 | 0.35 | 21.7 | 83 |
| ISOPHORONE | 2.02 | 1.8 | 1.7 | 2.3 | . | . | 1.93333 | 0.32 | 16.6 | 96 |
| ACENAPHTHYLENE | 2.02 | 1.3 | 1.4 | 1.7 | . | . | 1.46667 | 0.21 | 14.2 | 73 |
| ACENAPHTHENE | 2.01 | 1.2 | 1.2 | 1.5 | . | . | 1.3 | 0.17 | 13.3 | 65 |
| DIMETHYL PHTHALATE | 2.01 | 0.8 | 0.71 | 0.71 | . | . | 0.74 | 0.05 | 7.0 | 37 |
| 2,6-DINITROTOLUENE | 2.01 | 0.72 | 0.69 | 1.2 | . | . | 0.87 | 0.29 | 32.9 | 43 |
| FLUORENE | 2.01 | 1.1 | 1.1 | 1.3 | . | . | 1.16667 | 0.12 | 9.9 | 58 |
| 2,4-DINITROTOLUENE | 2.03 | 1.3 | 0.75 | 1.1 | . | . | 1.05 | 0.28 | 26.5 | 52 |
| 1,2-DIPHENYLHYDRAZINE | 2 | 1.8 | 1.6 | 2.3 | . | . | 1.9 | 0.36 | 19.0 | 95 |
| 4-CHLOROPHENYL PHENYL ETHER | 2.01 | 1.2 | 1.1 | 1.1 | . | . | 1.13333 | 0.06 | 5.1 | 56 |
| DIETHYL PHTHALATE | 2.02 | 1.8 | 1.6 | 2 | . | . | 1.8 | 0.20 | 11.1 | 89 |
| N-NITROSODIPHENYLAMINE | 2 | <0.5 | <0.5 | <0.5 | . | . | <0.5 | . | . | 25 |
| HEXACHLOROBENZENE | 1.61 | 1.2 | 0.62 | 0.85 | . | . | 0.89 | 0.29 | 32.8 | 55 |
| 4-BROMOPHENYL PHENYL ETHER | 2.07 | 1.8 | 2 | 1.7 | . | . | 1.83333 | 0.15 | 8.3 | 89 |
| PHENANTHRENE/ANTHRACENE | 4.04 | 3.4 | 3.9 | 3.4 | . | . | 3.56667 | 0.29 | 8.1 | 88 |
| DI-N-BUTYL PHTHALATE | 2.01 | 2 | 1.9 | 2.3 | . | . | 2.06667 | 0.21 | 10.1 | 103 |
| FLUORANTHENE | 2.01 | 2 | 1.8 | 1.8 | . | . | 1.86667 | 0.12 | 6.2 | 93 |
| PYRENE | 2.02 | 2.4 | 2.1 | 2.2 | . | . | 2.23333 | 0.15 | 6.8 | 111 |
| BENZIDINE | 2.01 | <0.5 | <0.5 | <0.5 | . | . | <0.5 | . | . | 25 |
| BUTYLBENZYL PHTHALATE | 2.02 | 1.9 | 1.6 | 2.1 | . | . | 1.86667 | 0.25 | 13.5 | 92 |
| BIS(2-ETHYLHEXYL) PHTHALATE | 2.01 | 1.8 | 1.6 | 2 | . | . | 1.8 | 0.20 | 11.1 | 16 |
| CHRYSENE/BENZO(A)ANTHRACENE | 2.02 | 1.8 | 1.6 | 1.8 | . | . | 1.73333 | 0.12 | 6.7 | 61 |
| 3,3'-DICHLOROBENZIDINE | 2.01 | 1.9 | 2 | 2.3 | . | . | 2.06667 | 0.21 | 10.1 | 103 |
| DI-N-OCTYL PHTHALATE | 2.03 | <0.5 | <0.5 | <0.5 | . | . | <0.5 | . | . | 25 |
| BENZO(B)/BENZO(K)FLUORANTHENES | 4.42 | 3 | 3.5 | 3.2 | . | . | 3.23333 | 0.25 | 7.8 | 58 |
| BENZO(A)PYRENE | 2.08 | 2.2 | 2.4 | 2.5 | . | . | 2.36667 | 0.15 | 6.5 | 60 |
| INDENO(1,2,3-CD)PYRENE | 0 | 0.6 | 0.53 | 0.51 | . | . | 0.546667 | 0.05 | 8.6 | . |
| DIBENZO(AH)ANTHRACENE | 0 | <0.5 | <0.5 | <0.5 | . | . | <0.5 | . | . | . |
| BENZO(GHI)PERYLENE | 2.02 | 1.1 | 1.3 | 1.7 | . | . | 1.36667 | 0.31 | 22.4 | 68 |

TABLE IV-41. BASE/NEUTRAL EXTRACTABLE ORGANICS DATA—
COKE OVEN BIOLOGICAL SLUDGE, SPIKE LEVEL II

| COMPOUND | ADDED | NO_1 | NO_2 | NO_3 | NO_4 | NO_5 | MEAN | S | RSD | RECOVERY |
|---|---|---|---|---|---|---|---|---|---|---|
| 1,3-DICHLOROBENZENE | 8.54 | 5.6 | 7 | 6.2 | . | . | 6.26667 | 0.70 | 11.2 | 73 |
| 1,4-DICHLOROBENZENE | 10.96 | 8.4 | 11 | 11 | . | . | 10.1333 | 1.50 | 14.8 | 92 |
| 1,2-DICHLOROBENZENE | 10.02 | 10 | 13 | 12 | . | . | 11.6667 | 1.53 | 13.1 | 116 |
| HEXACHLOROETHANE | 10.02 | 8.1 | 8.8 | 8.7 | . | . | 8.53333 | 0.38 | 4.4 | 85 |
| BIS(2-CHLOROETHYL) ETHER | 10 | 10 | 11 | 7 | . | . | 9.33333 | 2.08 | 22.3 | 93 |
| BIS(2-CHLOROISOPROPYL) ETHER | 10.02 | 5.7 | 6.6 | 3 | . | . | 5.1 | 1.87 | 36.7 | 51 |
| HEXACHLOROBUTADIENE | 10.23 | 11 | 11 | 12 | . | . | 11.3333 | 0.58 | 5.1 | 111 |
| NITROBENZENE | 10.03 | 4.3 | 5 | 3.3 | . | . | 4.2 | 0.85 | 20.3 | 42 |
| NAPHTHALENE | 10.01 | 8.7 | 9.2 | 6.4 | . | . | 8.1 | 1.49 | 18.4 | 81 |
| 1,2,4-TRICHLOROBENZENE | 11 | 1.6 | 1.2 | 1.3 | . | . | 1.36667 | 0.21 | 15.2 | 12 |
| BIS(2-CHLOROETHOXY)METHANE | 10.09 | 5.7 | 7 | 4.3 | . | . | 5.66667 | 1.35 | 23.8 | 56 |
| N-NITROSODI-N-PROPYLAMINE | 10.02 | 17 | 17 | 8.7 | . | . | 14.2333 | 4.79 | 33.7 | 142 |
| HEXACHLOROCYCLOPENTADIENE | 10.12 | 2 | 2 | 2.4 | . | . | 2.13333 | 0.23 | 10.8 | 21 |
| 2-CHLORONAPHTHALENE | 9.6 | 9.2 | 10 | 8.6 | . | . | 9.26667 | 0.70 | 7.6 | 97 |
| ISOPHORONE | 10.08 | 6.2 | 6.1 | 4 | . | . | 5.43333 | 1.24 | 22.9 | 54 |
| ACENAPHTHYLENE | 10.08 | 6.8 | 9.1 | 8.1 | . | . | 8 | 1.15 | 14.4 | 79 |
| ACENAPHTHENE | 10.06 | 8.9 | 9.2 | 8.2 | . | . | 8.76667 | 0.51 | 5.9 | 87 |
| DIMETHYL PHTHALATE | 10.07 | 7.2 | 8.4 | 7.9 | . | . | 7.83333 | 0.60 | 7.7 | 78 |
| 2,6-DINITROTOLUENE | 10.05 | 6.2 | 7 | 7.2 | . | . | 6.8 | 0.53 | 7.8 | 68 |
| FLUORENE | 10.04 | 7.8 | 11 | 9.9 | . | . | 9.56667 | 1.63 | 17.0 | 95 |
| 2,4-DINITROTOLUENE | 10.16 | 6.2 | 7 | 7.2 | . | . | 6.8 | 0.53 | 7.8 | 67 |
| 1,2-DIPHENYLHYDRAZINE | 10.02 | 4.9 | 4.8 | 4.7 | . | . | 4.8 | 0.10 | 2.1 | 48 |
| 4-CHLOROPHENYL PHENYL ETHER | 10.04 | 7.8 | 10 | 11 | . | . | 9.6 | 1.64 | 17.1 | 96 |
| DIETHYL PHTHALATE | 10.11 | 11 | 9.8 | 10 | . | . | 10.2667 | 0.64 | 6.3 | 102 |
| N-NITROSODIPHENYLAMINE | 10 | 5.2 | 5.8 | 6.1 | . | . | 5.7 | 0.46 | 8.0 | 57 |
| HEXACHLOROBENZENE | 8.07 | 6.8 | 8.7 | 9.3 | . | . | 8.26667 | 1.31 | 15.8 | 102 |
| 4-BROMOPHENYL PHENYL ETHER | 10.33 | 7.7 | 10 | 10 | . | . | 9.23333 | 1.33 | 14.4 | 89 |
| PHENANTHRENE/ANTHRACENE | 20.18 | 8.6 | 8.7 | 9.5 | . | . | 8.93333 | 0.49 | 5.5 | 44 |
| DI-N-BUTYL PHTHALATE | 10.05 | 8.8 | 8.3 | 9.8 | . | . | 8.96667 | 0.76 | 8.5 | 89 |
| FLUORANTHENE | 10.04 | 8.2 | 9.7 | 10 | . | . | 9.3 | 0.96 | 10.4 | 93 |
| PYRENE | 10.09 | 8.3 | 9.9 | 10 | . | . | 9.4 | 0.95 | 10.1 | 93 |
| BENZIDINE | 10.04 | 3 | 4.7 | 3.9 | . | . | 3.86667 | 0.85 | 22.0 | 39 |
| BUTYLBENZYL PHTHALATE | 10.09 | 4.4 | 3.8 | 4.5 | . | . | 4.23333 | 0.38 | 8.9 | 42 |
| BIS(2-ETHYLHEXYL) PHTHALATE | 10.06 | 3.3 | 3.4 | 3.3 | . | . | 3.33333 | 0.06 | 1.7 | 18 |
| CHRYSENE/BENZO(A)ANTHRACENE | 10.08 | 7.4 | 8.4 | 10 | . | . | 8.6 | 1.31 | 15.2 | 80 |
| 3,3'-DICHLOROBENZIDINE | 10.04 | 3 | 4.7 | 4.7 | . | . | 4.13333 | 0.98 | 23.7 | 41 |
| DI-N-OCTYL PHTHALATE | 10.13 | <0.5 | <0.5 | <0.5 | . | . | <0.5 | Z | . | 5 |
| BENZO(B)/BENZO(K)FLUORANTHENES | 22.11 | 34 | 44 | 43 | . | . | 40.3333 | 5.51 | 13.7 | 179 |
| BENZO(A)PYRENE | 10.4 | 18 | 24 | 22 | . | . | 21.3333 | 3.06 | 14.3 | 194 |
| INDENO(1,2,3-CD)PYRENE | 0 | <0.5 | <0.5 | 0.54 | . | . | <.513333 | 0.02 | 4.5 | . |
| DIBENZO(AH)ANTHRACENE | 0 | <0.5 | <0.5 | <0.5 | . | . | <0.5 | Z | . | . |
| BENZO(GHI)PERYLENE | 10.12 | 13 | 15 | 16 | . | . | 14.6667 | 1.53 | 10.4 | 145 |

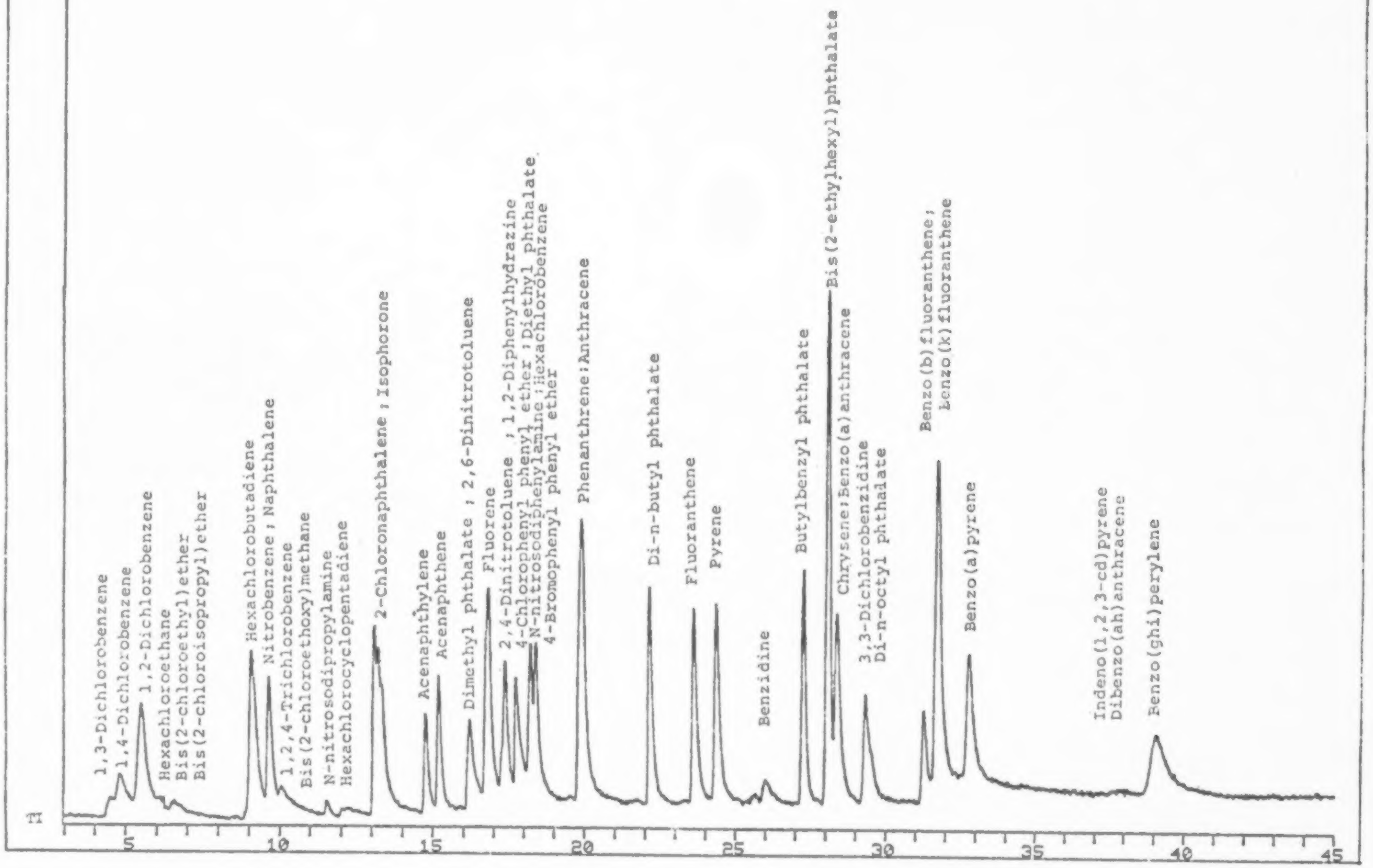


Figure IV-25. GC/MS chromatogram of base/neutral extractables—Coke Oven Biological Sludge, spiked

## TABLE IV-42. ACID EXTRACTABLE SEMIVOLATILE ORGANICS DATA—COKE OVEN BIOLOGICAL SLUDGE, SPIKE LEVEL 0

| COMPOUND | ADDED | NO_1 | NO_2 | NO_3 | NO_4 | NO_5 | MEAN | S | RSD | RECOVERY |
|---|---|---|---|---|---|---|---|---|---|---|
| 2-CHLOROPHENOL | 0 | 0 | 0 | 0 | . | . | 0 | Z | . | . |
| 2-NITROPHENOL | 0 | 0 | 0 | 0 | . | . | 0 | Z | . | . |
| PHENOL | 0 | <0.5 | <0.5 | <0.5 | . | . | <0.5 | Z | . | . |
| 2,4-DIMETHYLPHENOL | 0 | 0 | 0 | 0 | . | . | 0 | Z | . | . |
| 2,4-DICHLOROPHENOL | 0 | 0 | 0 | 0 | . | . | 0 | Z | . | . |
| 2,4,6-TRICHLOROPHENOL | 0 | 0 | 0 | 0 | . | . | 0 | Z | . | . |
| 4-CHLORO-3-METHYLPHENOL | 0 | 0 | 0 | 0 | . | . | 0 | Z | . | . |
| 2,4-DINITROPHENOL | 0 | 0 | 0 | 0 | . | . | 0 | Z | . | . |
| 4,6-DINITRO-O-CRESOL | 0 | 0 | 0 | 0 | . | . | 0 | Z | . | . |
| PENTACHLOROPHENOL | 0 | 0.92 | 3.3 | 2.3 | . | . | 2.17333 | 1.20 | 55.0 | . |
| 4-NITROPHENOL | 0 | 0 | 0 | 0 | . | . | 0 | Z | . | . |

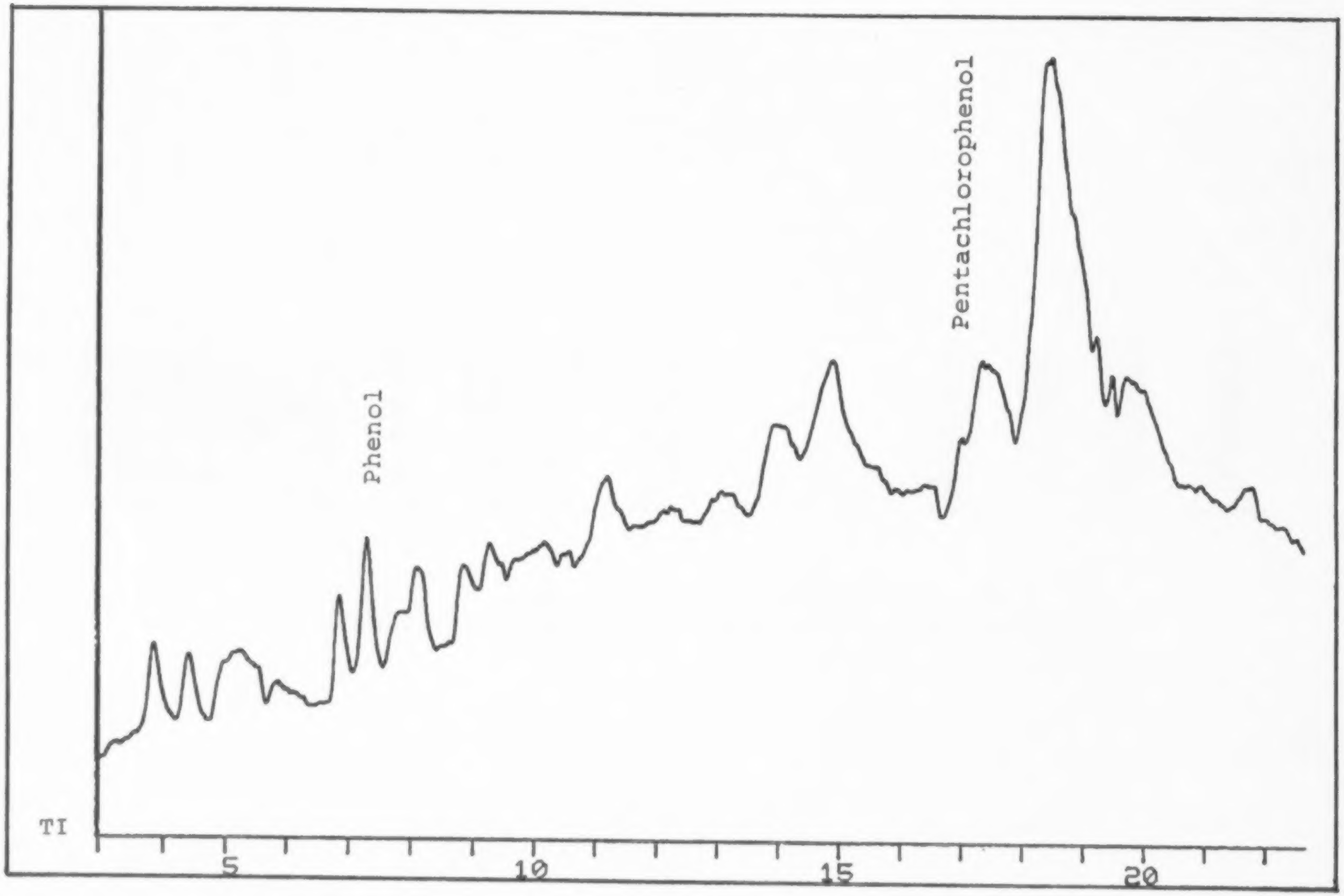


Figure IV-26. GC/MS chromatogram of acid extractables—Coke Oven Biological Sludge, unspiked

# TABLE IV-43. ACID EXTRACTABLE SEMIVOLATILE ORGANICS DATA—COKE OVEN BIOLOGICAL SLUDGE, SPIKE LEVEL I

| COMPOUND | ADDED | NO_1 | NO_2 | NO_3 | NO_4 | NO_5 | MEAN | S | RSD | RECOVERY |
|---|---|---|---|---|---|---|---|---|---|---|
| 2-CHLOROPHENOL | 4.01 | 4.6 | 6.3 | 4.6 | . | . | 5.16667 | 0.98 | 19.0 | 129 |
| 2-NITROPHENOL | 4.02 | 5.1 | 6.4 | 5 | . | . | 5.5 | 0.78 | 14.2 | 137 |
| PHENOL | 4 | 5.7 | 8.1 | 5.8 | . | . | 6.53333 | 1.36 | 20.8 | 151 |
| 2,4-DIMETHYLPHENOL | 4.02 | 1 | 1.2 | 0.78 | . | . | 0.993333 | 0.21 | 21.1 | 25 |
| 2,4-DICHLOROPHENOL | 4.04 | 5 | 6.2 | 4.9 | . | . | 5.36667 | 0.72 | 13.5 | 133 |
| 2,4,6-TRICHLOROPHENOL | 4.02 | 5 | 6.7 | 5.3 | . | . | 5.66667 | 0.91 | 16.0 | 141 |
| 4-CHLORO-3-METHYLPHENOL | 4.01 | 4.1 | 6.2 | 4.3 | . | . | 4.86667 | 1.16 | 23.8 | 121 |
| 2,4-DINITROPHENOL | 4 | 4.5 | 7.2 | 7.5 | . | . | 6.4 | 1.65 | 25.8 | 160 |
| 4,6-DINITRO-O-CRESOL | 4.02 | 3.8 | 6.3 | 5.6 | . | . | 5.23333 | 1.29 | 24.6 | 130 |
| PENTACHLOROPHENOL | 4.01 | 4.1 | 5.3 | 4.4 | . | . | 4.6 | 0.62 | 13.6 | 61 |
| 4-NITROPHENOL | 4.01 | 5 | 6.8 | 5.5 | . | . | 5.76667 | 0.93 | 16.1 | 144 |

## TABLE IV-44. ACID EXTRACTABLE SEMIVOLATILE ORGANICS DATA— COKE OVEN BIOLOGICAL SLUDGE, SPIKE LEVEL II

| COMPOUND | ADDED | NO_1 | NO_2 | NO_3 | NO_4 | NO_5 | MEAN | S | RSD | RECOVERY |
|---|---|---|---|---|---|---|---|---|---|---|
| 2-CHLOROPHENOL | 10.03 | 9.7 | 9.5 | 9.3 | . | . | 9.5 | 0.20 | 2.1 | 95 |
| 2-NITROPHENOL | 10.05 | 14 | 12 | 13 | . | . | 13 | 1.00 | 7.7 | 129 |
| PHENOL | 10 | 8 | 9.6 | 9.9 | . | . | 9.16667 | 1.02 | 11.1 | 87 |
| 2,4-DIMETHYLPHENOL | 10.04 | 1.8 | 2.3 | 1.1 | . | . | 1.73333 | 0.60 | 34.8 | 17 |
| 2,4-DICHLOROPHENOL | 10.11 | 13 | 12 | 11 | . | . | 12 | 1.00 | 8.3 | 119 |
| 2,4,6-TRICHLOROPHENOL | 10.06 | 9 | 11 | 9.6 | . | . | 9.86667 | 1.03 | 10.4 | 98 |
| 4-CHLORO-3-METHYLPHENOL | 10.02 | 9.7 | 11 | 8.2 | . | . | 9.63333 | 1.40 | 14.5 | 96 |
| 2,4-DINITROPHENOL | 10 | 9 | 7.8 | 5.1 | . | . | 7.3 | 2.00 | 27.4 | 73 |
| 4,6-DINITRU-U-CRESOL | 10.04 | 8.7 | 6 | 7.2 | . | . | 7.3 | 1.35 | 18.5 | 73 |
| PENTACHLOROPHENOL | 10.02 | 5.1 | 6.9 | 6.5 | . | . | 6.16667 | 0.95 | 15.3 | 40 |
| 4-NITROPHENOL | 10.02 | 6.8 | 10 | 11 | . | . | 9.26667 | 2.19 | 23.7 | 92 |

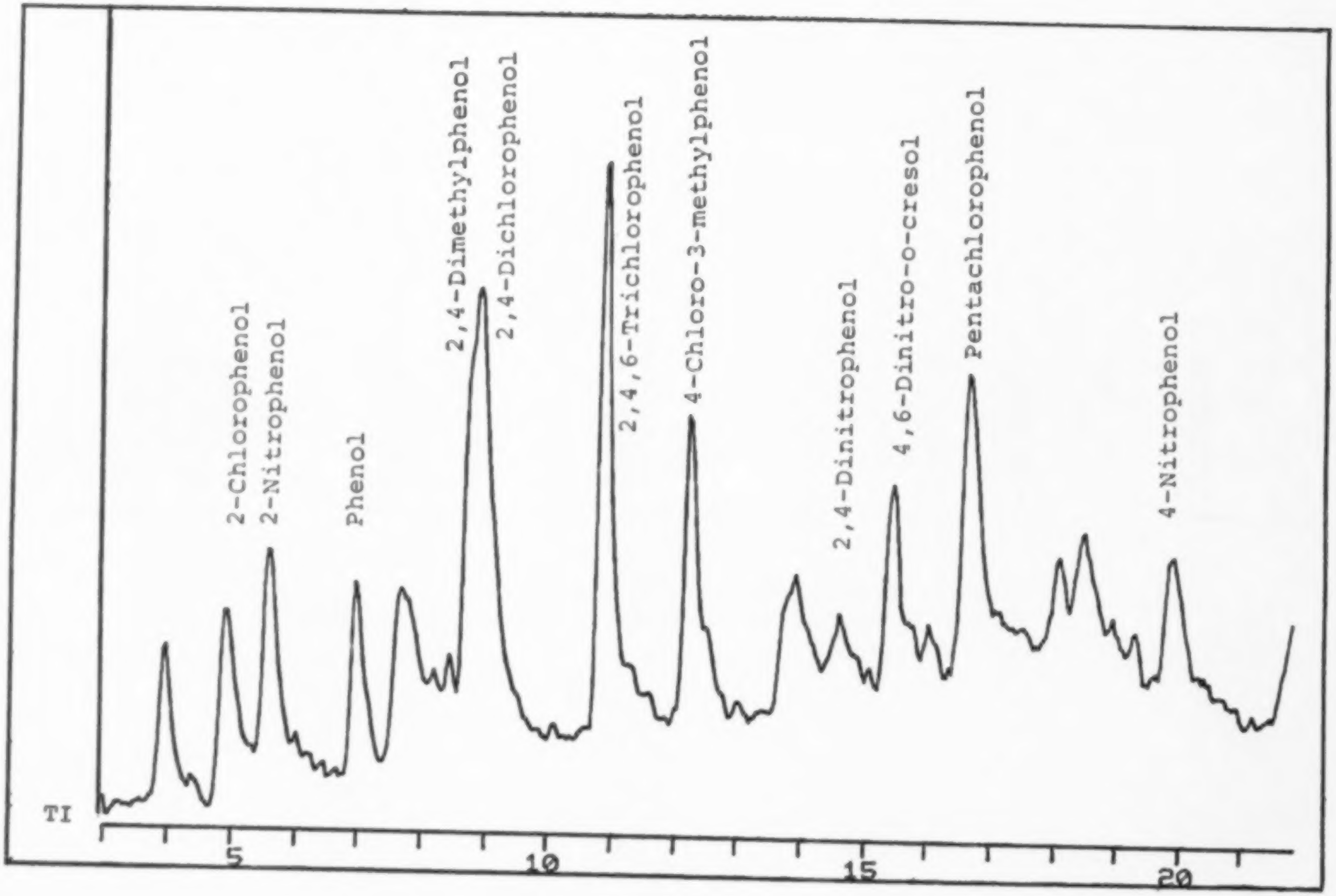


Figure IV-27. GC/MS chromatogram of acid extractables—Coke Oven Biological Sludge, spiked

## G. Electroplating Sludge

## TABLE IV-45. METALS DATA—
## ELECTROPLATING SLUDGE, SPIKE LEVEL 0

| ELEMENT | ADDED | NO_1 | NO_2 | NO_3 | NO_4 | NO_5 | MEAN | S | RSD | RECOVERY |
|---|---|---|---|---|---|---|---|---|---|---|
| ANTIMONY | 0 | 6.5 | 7.4 | 6.8 | . | . | 6.9 | 0.46 | 6.6 | . |
| ARSENIC | 0 | 2.8 | 3 | 3.2 | . | . | 3 | 0.20 | 6.7 | . |
| BERYLLIUM | 0 | <0.3 | <0.3 | <0.3 | . | . | <0.3 | Z | . | . |
| CADMIUM | 0 | 0.71 | 0.65 | 0.59 | . | . | 0.65 | 0.06 | 9.2 | . |
| CHROMIUM | 0 | 2480 | 2470 | 2550 | . | . | 2500 | 43.59 | 1.7 | . |
| COPPER | 0 | 24800 | 25500 | 25000 | . | . | 25100 | 360.56 | 1.4 | . |
| LEAD | 0 | 1220 | 1260 | 1300 | . | . | 1260 | 40.00 | 3.2 | . |
| MERCURY | 0 | <0.02 | <0.02 | <0.02 | . | . | <0.02 | Z | . | . |
| NICKEL | 0 | 3900 | 4200 | 4000 | . | . | 4033.33 | 152.75 | 3.8 | . |
| SELENIUM | 0 | <1 | <1 | <1 | . | . | <1 | Z | . | . |
| SILVER | 0 | <0.5 | <0.5 | <0.5 | . | . | <0.5 | Z | . | . |
| THALLIUM | 0 | <1 | <1 | <1 | . | . | <1 | Z | . | . |
| ZINC | 0 | 1900 | 1890 | 1930 | . | . | 1906.67 | 20.82 | 1.1 | . |

## TABLE IV-46. METALS DATA— ELECTROPLATING SLUDGE, SPIKE LEVEL I

| ELEMENT | ADDED | NO_1 | NO_2 | NO_3 | NO_4 | NO_5 | MEAN | S | RSD | RECOVERY |
|---|---|---|---|---|---|---|---|---|---|---|
| ANTIMONY | 16.3 | 16 | 15 | 16 | . | . | 15.6667 | 0.58 | 3.7 | 54 |
| ARSENIC | 16.3 | 14 | 13 | 14 | . | . | 13.6667 | 0.58 | 4.2 | 65 |
| BERYLLIUM | 16.3 | 11 | 10 | 11 | . | . | 10.6667 | 0.58 | 5.4 | 64 |
| CADMIUM | 16.3 | 17 | 16 | 16 | . | . | 16.3333 | 0.58 | 3.5 | 96 |
| CHROMIUM | 651 | 3250 | 3170 | 3230 | . | . | 3216.67 | 41.63 | 1.3 | 110 |
| COPPER | 0 | 25000 | 25900 | 24100 | . | . | 25000 | 900.00 | 3.6 | . |
| LEAD | 651 | 1900 | 1890 | 1930 | . | . | 1906.67 | 20.82 | 1.1 | 99 |
| MERCURY | 16.3 | 14 | 13 | 13 | . | . | 13.3333 | 0.58 | 4.3 | 82 |
| NICKEL | 1950 | 6160 | 5810 | 5240 | . | . | 5736.67 | 464.36 | 8.1 | 87 |
| SELENIUM | 16.3 | 9.1 | 11 | 9.1 | . | . | 9.73333 | 1.10 | 11.3 | 54 |
| SILVER | 16.3 | <0.5 | <0.5 | <0.5 | . | . | <0.5 | Z | . | 0 |
| THALLIUM | 33 | 28 | 26 | 35 | . | . | 29.6667 | 4.73 | 15.9 | 87 |
| ZINC | 977 | 3010 | 2960 | 2990 | . | . | 2986.67 | 25.17 | 0.8 | 111 |

## H. Electric Furnace Baghouse Dust

TABLE IV-47. METALS DATA—
ELECTRIC FURNACE BAGHOUSE DUST, SPIKE LEVEL 0

| ELEMENT | ADDED | NO_1 | NO_2 | NO_3 | NO_4 | NO_5 | MEAN | S | RSD | RECOVERY |
|---|---|---|---|---|---|---|---|---|---|---|
| ANTIMONY | 0 | 9.4 | 8.1 | 8.6 | . | . | 8.7 | 0.66 | 7.5 | . |
| ARSENIC | 0 | 38 | 32 | 32 | . | . | 34 | 3.46 | 10.2 | . |
| BERYLLIUM | 0 | <0.2 | <0.2 | <0.2 | . | . | <0.2 | Z | . | . |
| CADMIUM | 0 | 9.4 | 8.1 | 10 | . | . | 9.16667 | 0.97 | 10.6 | . |
| CHRCMIUM | 0 | 1670 | 1610 | 1520 | . | . | 1600 | 75.50 | 4.7 | . |
| COPPER | 0 | 424 | 416 | 429 | . | . | 423 | 6.56 | 1.6 | . |
| LEAD | 0 | 1400 | 1280 | 1280 | . | . | 1320 | 69.28 | 5.2 | . |
| MERCURY | 0 | 1.4 | 1.2 | 1.2 | . | . | 1.26667 | 0.12 | 9.1 | . |
| NICKEL | 0 | 525 | 547 | 571 | . | . | 547.667 | 23.01 | 4.2 | . |
| SELENIUM | 0 | <1 | <1 | <1 | . | . | <1 | Z | . | . |
| SILVER | 0 | 0.8 | 0.8 | 0.9 | . | . | 0.833333 | 0.06 | 6.9 | . |
| THALLIUM | 0 | <1 | <1 | <1 | . | . | <1 | Z | . | . |
| ZINC | 0 | 73500 | 65300 | 65500 | . | . | 68100 | 4677.61 | 6.9 | . |

TABLE IV-48. METALS DATA—
ELECTRIC FURNACE BAGHOUSE DUST, SPIKE LEVEL I

| ELEMENT | ADDED | NO_1 | NO_2 | NO_3 | NO_4 | NO_5 | MEAN | S | RSD | RECOVERY |
|---|---|---|---|---|---|---|---|---|---|---|
| ANTIMONY | 12.5 | 17 | 19 | 18 | . | . | 18 | 1.00 | 5.6 | 74 |
| ARSENIC | 12.5 | 41 | 37 | 34 | . | . | 37.3333 | 3.51 | 9.4 | 27 |
| BERYLLIUM | 12.5 | 11 | 11 | 11 | . | . | 11 | 0.00 | 0.0 | 86 |
| CADMIUM | 12.5 | 18 | 18 | 16 | . | . | 17.3333 | 1.15 | 6.7 | 65 |
| CHROMIUM | 125 | 1760 | 1710 | 1760 | . | . | 1743.33 | 28.87 | 1.7 | 115 |
| COPPER | 125 | 532 | 548 | 543 | . | . | 541 | 8.19 | 1.5 | 94 |
| LEAD | 500 | 1830 | 1620 | 1840 | . | . | 1763.33 | 124.23 | 7.0 | 89 |
| MERCURY | 12.5 | 13 | 18 | 15 | . | . | 15.3333 | 2.52 | 16.4 | 113 |
| NICKEL | 250 | 812 | 784 | 786 | . | . | 794 | 15.62 | 2.0 | 99 |
| SELENIUM | 12.5 | 8 | 9.5 | 8 | . | . | 8.5 | 0.87 | 10.2 | 60 |
| SILVER | 12.5 | 0.9 | 0.6 | 0.9 | . | . | 0.8 | 0.17 | 21.7 | N |
| THALLIUM | 25 | 19 | 18 | 19 | . | . | 18.6667 | 0.58 | 3.1 | 71 |
| ZINC | 0 | 68300 | 64100 | 61600 | . | . | 64666.7 | 3385.75 | 5.2 | . |

# Appendix V. Text of Methods for Total Content

## A. Proposed Method for Purgeable Organics in Residual Waste

# PROPOSED METHOD FOR PURGEABLE ORGANICS IN RESIDUAL WASTE
(January 1981)

## 1. Scope and Application

1.1 Scope—This method is used for the determination of purgeable organic compounds. A selected list of compounds that may be determined by this method is provided in Table V-1.

1.2 Application—The method is applicable to the measurement of purgeable organics in commercial residual wastes. It is designed to be used to meet the survey requirements of the Office of Solid Waste (OSW). The method uses a purge-and-trap apparatus for isolation and concentration of purgeable organic compounds and a GC/MS system for qualitative and quantitative determination of these compounds. Because of the diverse nature and complexity of residual waste, the method described below is recognized as broad in scope; nevertheless, it was written with a priority of simplicity in mind. Of course, no single method is applicable to all waste matrices and some method modification may be necessary for unusual waste types.

## 2. Summary of Method

2.1 An appropriate weight of sludge (determined by preliminary screening of the extractable organic content of the sample) is diluted to 10 mL with organic-free water. The diluted sample is purged at room temperature (∿25 °C) with an inert gas for 12 min. Water-insoluble compounds boiling below 200 °C are transferred from the aqueous phase to the gaseous phase. The gaseous phase is passed through a sorbent trap where the organic compounds are concentrated. The contents of the trap are then desorbed into the GC/MS by heating and backflushing the trap. Because variations in recovery efficiencies for the individual purgeable organics can be affected by the sample matrices, extensive quality control is required for accurate measurements. The total analysis time is less than 1 h. This method is recommended for use only by experienced analysts or by experienced technicians under the close supervision of a qualified analyst.

## 3. Apparatus and Reagents

3.1 For sample preparation.

3.1.1 Purge-and-trap system—Assemble the system depicted in Figures V-1 and V-2. If desired, a commercial version of this system, such as the Tekmar Liquid Sample Concentrator Model LSC-1, or its equivalent, may be used.

Details of the purging device in Figures V-1 and V-2 are shown in Figure V-3. Details of the trap in Figures V-1 and V-2 are shown in Figure V-4. The trap may be purchased commercially (Supelco 2-0293) or packed with the individual elements in the following order: Place the glass wool plug in the inlet end of the trap. Follow this with the 3% OV-1, Tenax-GC, silica gel, activated charcoal, and the second glass wool plug. This order must be followed if the trap is to perform properly. Reversing the packing order (i.e., placing the charcoal in the trap first) will cause the silica gel and Tenax-GC layers to become contaminated with charcoal dust and will cause poor desorption efficiencies. Install the trap so that the effluent from the purging device enters the Tenax end of the trap.

3.1.2 Glassware.

a. Screw-cap vials—40 mL with Teflon-lined caps.

b. Graduated pipets—1, 5, and 10 mL with tips cut off. Disposable macropipets such as Fisher No. 21-164-4E may also be used.

c. Volumetric flasks—10 mL.

3.1.3 Analytical balance.

3.1.4 Roller mill and 1/8 in. stainless steel ball bearings (or equivalent mixing apparatus).

3.1.5 Catalytic gas purifier (optional).

3.1.6 Purging gas—helium or nitrogen, high-purity grade.

3.1.7 Syringes—10 µL, 100 µL, and 5 mL; gas tight for quality control spiking.

3.1.8 Water free of purgeable organics (see Section 5).

3.1.9 Packing materials for sorbent trap.

a. 3% OV-1 on Chromosorb W, 100/110 mesh.

b. Tenax-GC—60/80 mesh.

c. Silica gel—Davison Grade 15, 35/60 mesh or equivalent.

d. Coconut charcoal—Barnebey-Cheney #CA-580-26, 26 mesh or equivalent.

3.1.10 Glass wool—Cleaned by thorough rinsing with hexane, dried in a 110 °C oven, and stored in a hexane-rinsed glass jar with a Teflon-lined cap.

3.2 For quantitation and identification.

3.2.1 Gas chromatograph/mass spectrometer/data system—Capable of scanning from 35 to 350 a.m.u. every 4 sec or less at 70 volts (nominal) and producing a recognizable mass spectrum (background corrected) at unit resolution from 50 ng of decafluorotriphenylphosphine (DFTPP) when the sample is introduced through the inlet of the chromatograph (Reference 1). Evaluate the system performance each day by examining the spectrum of DFTPP or p-bromofluorobenze (BFB). Inject 50 ng of DFTPP and compare with the performance criteria listed in Table V-2. To use BFB, inject 50 ng of BFB and compare with the performance criteria listed in Table V-3. The mass spectrometer must be interfaced with a gas chromatograph equipped with an injector system designed for all-glass on-column injection on packed columns or, alternatively, equipped with a capillary injector system designed for splitless injection and glass capillary columns. All sections of the transfer lines must be glass or glass-lined and must be deactivated. (Use Sylon-CT, Supelco, Inc., or equivalent to deactivate.) Note: Systems utilizing a jet separator for the GC effluent are required since membrane separators may cause loss of sensitivity to small molecules.

3.2.2 The GC/MS should be fitted with a 6-ft glass column (1/4 in. OD x 2 mm ID) packed with 1% SP 1000 on Carbopack B, 60/80 mesh, preceded by a 5-cm precolumn packed with 1% SP-1000 on Chromosorb W, 60/80 mesh. The precolumn is only necessary during conditioning. An alternate packing to the SP-1000 on Carbopack B is 0.2% Carbowax 1500 on Carbopack C, 60/80 mesh. An 8-ft stainless steel column can be substituted for the 6-ft glass column.

3.2.3 A computer system must be interfaced to the mass spectrometer to allow acquisition of continuous mass scans for the duration of the chromatographic program. The computer system should also be equipped with mass storage devices for saving all data from GC/MS runs. There must be computer software available to allow searching any GC/MS run for specific ions and plotting the intensity of the ions with respect to time or scan number. The ability to integrate the area under any specific ion plot peak is essential for quantification.

3.2.4 Reference materials—These materials consist of assayed, authentic samples of all of the compounds to be determined and in addition the following standards—(1) internal standards (Section 6.3), (2) surrogate standards (Section 6.4), (3) decafluorotriphenylphosphine (a calibration standard for the MS), (4) *p*-bromofluorobenzene (also a calibration standard for the MS).

## 4. Sample Collection and Preservation

4.1 Container preparation—Samples must be collected in 40-mL screw-cap vials with zero head space and sealed with Teflon-lined caps (larger wide-mouth bottles with Teflon-lined caps may be used). Wash all sample bottles in detergent before use; rinse with tap water and finally with distilled water. Rinse the Teflon seals with distilled water and allow the bottles and seals to air dry at room temperature. Heat in a 100 °C oven for 1 h; then allow to cool in an area known to be free of organics. Do not heat the Teflon seals for extended periods of time (more than 1 h) because the silicone layer slowly degrades at 100 °C.

4.2 Sampling—Samples are collected at discrete time intervals by grab sampling. When data for relatively long time intervals, such as 24 h, are necessary or desirable, compositing of several grab samples must be performed. To do this, collect six grab samples (i.e., one 40-mL grab sample collected every 4 h over a 24-h period) and composite the six grab samples.

4.3 Preservation—As a general guideline, ice samples immediately after collection, refrigerate at 4 °C, and purge within 10 days.

4.4 Sample handling—The chilled samples are mixed by gentle swirling in a 250-mL wide-mouth flask. (Vigorous mixing must be avoided to prevent analyte losses.) Analysis should be performed immediately after compositing. When this is impossible or sample material is to be retained for future reference, aliquots of the composite sample should be returned to cleaned vials with zero head space and stored at 4 °C.

## 5. Preparation of Reagent Water Free of Purgeable Organics

Reagent water is generated by passing tap water through a carbon filter bed containing about 1 lb of activated carbon. The water is purged overnight with prepurified nitrogen. A Millipore Super-Q Water System or its equivalent may be used to generate reagent water. Alternatively, reagent water can be prepared by boiling distilled water for 15 min. Transfer the water while it is still hot to a glass-stoppered bottle. Cool to room temperature. Test the reagent water daily by analyzing it according to the method in Section 9.1.

## 6. Preparation of Standards

The following protocol assumes the preparation of stock solutions from the pure compounds. Commercially available mixed stock solutions, if they are shown to be satisfactory, may be used instead.

6.1 Analytical standards (solutions of authentic samples of the compounds to be determined)—Prepare standard stock solutions of liquid compounds (at approximately 20 μg/μL) by adding, from a 100-μL syringe, 1 to 2 drops (~0.2 mL) of the 99+% pure reference standard to methyl alcohol (9 mL) contained in a tared 10-mL volumetric flask (weighed to nearest 0.1 mg). Add the component so that the drops fall into the alcohol and do not make contact with the neck of the flask. Prepare standards of gaseous compounds (e.g., vinyl chloride) in a similar manner using a 50-mL valved gas-tight syringe with a 2-in. needle. Fill the syringe with the gaseous compound. Weigh a 10-mL volumetric flask containing 9 mL of methyl alcohol to the nearest 0.1 mg. Lower the syringe needle to about 5 mm below the methyl alcohol meniscus and slowly inject the standard into the flask. The gas rapidly dissolves in the methyl alcohol. Reweigh the flask, and use the weight gain to calculate the concentration of the standard. Dilute the volume, mix, and transfer to a 10-mL screw-cap vial and seal with a Teflon-lined cap. Gas stock standards are generally stable for at least 1 wk when maintained below 0 °C. Stock standards of compounds that boil above room temperature, with the exception of 2-chloroethyl vinyl ether, are generally stable for at least 4 wk when stored at 4 °C. (Safety caution: Because of the toxicity of most organohalides, precaution should be taken by weighing and making dilutions of standards in a fume hood.)

6.2 Solution for sample spiking (a solution of authentic samples of the compounds to be determined)—Prepare the spiking solution from appropriate stock solutions in accordance with the spiking procedures given in the QA/QC protocol.

6.3 Internal standards spiking solution—From a neat compound, weigh out 5 mg of $d_8$-toluene or $d_6$-benzene in a 50-mL volumetric flask with 45 mL of methyl alcohol in the container; mix and dilute to volume (0.10 μg/μL). Add 5 to 10 μL (depending on background interference) of this internal standard spiking solution to every sample and analytical standard. The 50-mL volume of the internal standard solution can be transferred to 10-mL serum vials and sealed with Teflon-lined septa and crimp-on caps. Keep the internal standard solution below 4 °C until use. Let the vial reach room temperature and rinse off the outer surface before removing aliquots.

6.4 Surrogate standard spiking solution—From stock solutions prepared as in Section 6.1, add 1 mg each of bromochloromethane and 1,4-dichlorobutane to 45 mL of reagent water contained in a 50-mL volumetric flask; mix and dilute to volume. Add 10 µL of this surrogate spiking solution to every sample and analytical standard. Prepare a fresh surrogate standard spiking solution on a weekly basis. Surrogate standards can be attained commercially (Supelco 4-8823).

## 7. Sample Preparation and Purging

7.1 Weigh an appropriate sample into a pretared 10- to 15-mL Teflon-lined, screw-capped vial.

Dilute the sample to 10 mL with reagent water. Transfer the total sample or an aliquot of the purge device using a syringe with an 1/8-in. gauge Teflon needle. Seal the sample in the purge device. Add an appropriate volume of surrogate and internal standard solutions. With a gas flow of 40 mL/min (nitrogen or helium), purge the sample 12 min at room temperature. If the sample foams excessively, stop purging. Place a glass wool plug in the top of purge tube to disperse the foam, seal the purge device, and continue to purge (with excessive foam, smaller sample sizes may be desired).

7.2 Weigh a second sample into a vial. Add the method recovery spiking solution (Section 6.2) underneath the surface of the sample; also add the surrogate and internal standard solutions. Analyze as in Section 7.1.

## 8. Analysis of the Sample Purge

8.1 Heat the trap to 200 °C. Backflush it for 4 min in the desorb mode into the gas chromatograph. Analyze the sample purge by GC/MS using the 1% SP-1000 on Carbopack B column operated with a helium carrier gas flow of 30 mL/min. Hold at 50 °C for 4 min, and then program at 10 °C/min to 220 °C. Hold at this temperature until all compounds have been eluted. The MS should be repetitively scanned over the range m/e equal to 20 to 275 in 4 sec or less. (A representative chromatogram is shown in Figure V-5.)

8.2 The purging device must be thoroughly rinsed with reagent water between successive samples. Thoroughly clean the purging device by flushing 10 mL of water into the purge device and removing the water; do this several times before leaving the desorb mode. Upon returning to the purge mode, the trap must be conditioned at 200 °C with gas flow for 10 min between successive samples. Let the trap cool 10 min before the next sample is added. For dirty samples, longer conditioning may be required.

## 9. Analytical Quality Assurance

9.1 System blanks—Analyze daily a reagent water blank spiked with 50 ng of *p*-bromofluorobenzene prior to the analysis of samples. Check the spectrum obtained for *p*-bromofluorobenzene and adjust the MS tuning parameters as required to meet the ion abundance criteria specified in Table V-3. The intensities of Extracted Ion Current Profiles (EICP's) for the internal standards give an overall check of the system sensitivity.

9.2 Analytical standards—Analyze standard solutions of low and high concentrations of each day that sample analyses are conducted. Response factor data obtained from these standards are used to determine the concentrations of compounds identified in the samples.

9.3 For the first sampling of each site within an industrial subcategory, the method recovery must be determined.

9.4 Spiked and duplicate samples—Analyze spiked and duplicate samples according to procedures in the QA/QC protocol.

9.5 *All* samples and analytical standards shall be spiked with internal standard and surrogate spiking solutions at concentrations of the same order of magnitude as the concentrations of spike recovery compounds.

## 10. Data Handling

Using the characteristic retention times and ions listed in Tables V-4 and V-5, obtain Extracted Ion Current Profiles (EICP's) of the characteristic ions for each compound. Verify the presence of compounds of interest on the basis of retention times and intensities of the characteristic ions. Calculate the concentrations of compounds identified by comparing the areas of the primary (highest abundance) ion peaks with the areas of the corresponding standard peaks. If the sample matrix produces a significant interference with the primary ion EICP, a secondary ion plot may be used for quantitation. Calculate the concentrations in the sample as follows:

$$\left(\frac{A}{B}\right)\left(\frac{B_{IS}}{A_{IS}}\right)\left(\frac{N}{W}\right) = \mu g/g \text{ of pollutant in wet sludge.}$$

where:

$A$ = area of peak in the sample.

$B$ = area of peak in the analytical standard,

$A_{IS}$ = area of internal standard peak in the sample.

$B_{IS}$ = area of internal standard peak in the analytical standard.

$N$ = micrograms in the analytical standard.

$W$ = weight of wet sludge analyzed (g).

## 11. References

11.1 "Reference Compound to Calibrate Ion Abundance Measurement in Gas Chromatography—Mass Spectrometry Systems," J. W. Eichelberger, L. E. Harris, and W. L. Budde, Anal. Chem. 47, 995-1000 (1975).

11.2 "Development of Analytical Test Procedures for the Measurement of Organic Priority Pollutants in Sludge and Sediments," Midwest Research Institute, Final Report EPA Contract No. 68-03-2695, June 26, 1979.

11.3 "Method for Purgeable Organics," (Unpublished), Personal Communication with MERL-EPA, Cincinnati, Ohio 45268, March 1980.

11.4 "Interim Methods for the Measurement of Organic Priority Pollutants in Sludges," U. S. Environmental Protection Agency Environmental Monitoring and Support Laboratory, Cincinnati, Ohio 45268, September 1979.

## TABLE V-1. SELECTED PURGEABLE ORGANICS DETECTABLE WITH THE PURGE-AND-TRAP METHOD

A. Fluoro-, Chloro-, and Bromomethanes

Methylene chloride (dichloromethane)
Chloroform (trichloromethane)
Carbon tetrachloride (tetrachloromethane)
Bromoform (tribromomethane)
Chlorodibromomethane
Bromodichloromethane
Trichlorofluoromethane

B. Chloroethanes

1,1-Dichloroethane
1,2-Dichloroethane
1,1,1-Trichloroethane
1,1,2-Trichloroethane
1,1,2,2-Tetrachloroethane

C. Benzenes and Chlorobenzenes

Benzene
Chlorobenzene
Toluene
Ethylbenzene

D. Chloroethylenes, -propanes, and -propenes

1,1-Dichloroethylene (1,1-dichloroethene)
Trans-1,2-dichloroethylene
  (Trans-1,2-dichloroethene)
Trichloroethylene (trichloroethene)
Tetrachloroethylene (tetrachloroethene)
1,2-Dichloropropane
Trans-1,3-dichloropropene
Cis-1,3-dichloropropene

TABLE V-2. IONS AND ION ABUNDANCE CRITERIA OF DECAFLUOROTRIPHENYLPHOSPHINE (DFTPP)

| M/E | Ion abundance criteria |
|---|---|
| 51 | 30 to 60% of mass 198 |
| 68 | Less than 2% of mass 69 |
| 70 | Less than 2% of mass 69 |
| 127 | 40 to 60% of mass 198 |
| 197 | Less than 1% of mass 198 |
| 198 | Base peak, 100% relative abundance |
| 199 | 5 to 9% of mass 198 |
| 275 | 10 to 30% of mass 198 |
| 365 | Greater than 1% of mass 198 |
| 441 | Present but less than mass 443 |
| 442 | Greater than 40% of mass 198 |
| 443 | 17 to 23% of mass 442 |

TABLE V-3. IONS AND ION ABUNDANCE CRITERIA OF p-BROMOFLUOROBENZENE (BFB)

| M/E | Ion abundance criteria |
|---|---|
| 50 | 20 to 40% of mass 95 |
| 75 | 55 to 75% of mass 95 |
| 95 | base peak, 100% relative abundance |
| 174 | 75 to 98% of mass 95 |
| 175 | 5 to 9% of mass 174 |
| 176 | 75 to 98% of base peak and 93 to 99% of mass 174 |
| 177 | 0 to 5% of mass 176 |

TABLE V-4. ELUTION ORDER AND DETECTABILITIES OF SELECTED PURGEABLE ORGANICS BY THE GC/MS METHOD[a]

| Compound | Relative retention time[b] | Limit of detection[c] | |
|---|---|---|---|
| | | ng injected | μg/g of sample |
| Methylene chloride | 0.38 | 10 | 0.1 |
| Trichlorofluoromethane | 0.39 | 10 | 0.1 |
| 1,1-Dichloroethylene | 0.46 | 10 | 0.1 |
| Bromochloromethane | 0.47 | 10 | 0.1 |
| 1,1-Dichloroethane | 0.48 | 10 | 0.1 |
| Trans-1,2-dichloroethylene | 0.52 | 10 | 0.1 |
| Chloroform | 0.53 | 10 | 0.1 |
| 1,2-Dichloroethane | 0.56 | 10 | 0.1 |
| 1,1,1-Trichloroethane | 0.61 | 10 | 0.1 |
| Carbon tetrachloride | 0.62 | 10 | 0.1 |
| Bromodichloromethane | 0.64 | 10 | 0.1 |
| 1,2-Dichloropropane | 0.69 | 10 | 0.1 |
| Trans-1,3-dichloropropene | 0.70 | 10 | 0.1 |
| Trichloroethylene | 0.73 | 10 | 0.1 |
| Dibromochloromethane | 0.75 | 10 | 0.1 |
| Cis-1,3-dichloropropene | 0.76 | 10 | 0.1 |
| 1,1,2-Trichloroethane | 0.76 | 10 | 0.1 |
| Benzene | 0.76 | 10 | 0.1 |
| 2-Bromo-1-chloropropane | 0.80 | 10 | 0.1 |
| Bromoform | 0.86 | 10 | 0.1 |
| 1,1,2,2-Tetrachloroethene | 0.95 | 10 | 0.1 |
| 1,1,2,2-Tetrachloroethane | 0.95 | 10 | 0.1 |
| 1,4-Dichlorobutane | 0.96 | 10 | 0.1 |
| Toluene | 1.00 | 10 | 0.1 |
| Chlorobenzene | 1.05 | 10 | 0.1 |
| Ethylbenzene | 1.15 | 10 | 0.1 |

a. These data were obtained under the following conditions: GC column, 6 ft x 2 mm ID glass column packed with 1% SP-1000 coated on Carbopack B, 60/80 mesh; carrier flow—30 mL/min; temperature program—50 °C held for 4 min, programmed 10 °C/min to 220 °C and held until all compounds have been eluted.

b. Relative to Toluene at 22.1 min.

c. This is a minimum level at which the entire analytical system must give recognizable mass spectra (background corrected) and acceptable calibration points. The overall level of detection is based on purging 0.1 gram of sample and desorbing total volatiles to the MS system. This level may be difficult to achieve depending on the nature of the sample.

## TABLE V-5. CHARACTERISTIC IONS OF PURGEABLE ORGANICS

| Compound | EI ions (relative intensity) | Ion used to quantify |
|---|---|---|
| Methylene chloride | 49(100); 51 (33); 84 (86); 86 (55) | 84 |
| Trichlorofluromethane | 101(100); 103 (66); | 101 |
| 1,1,-Dichloroethylene | 61(100); 96 (80); 98 (53) | 96 |
| Bromochloromethane | 49(100); 130 (88); 128 (70); 51 (33) | 128 |
| 1,1-Dichloroethane | 63(100); 65 (33); 83 (8); 98 (7); 100(4) | 63 |
| Trans-1,2-dichloroethylene | 64(100); 96 (90); 98 (57) | 96 |
| Chloroform | 83(100); 85 (66) | 83 |
| 1,2-Dichloroethane | 62(100); 64 (33); 98 (23); 100 (15) | 98 |
| 1,1,1-Trichloroethane | 97(100); 99 (66); 117 (17); 119 (16) | 97 |
| Carbon tetrachloride | 117(100); 119 (96), 121 (30) | 117 |
| Bromodichloromethane | 83(100); 85 (66); 127 (13); 129 (17) | 127 |
| 1,2-Dichloropropane | 63(100); 65 (33); 112 (4); 114 (3) | 112 |
| Trans-1,3-dichloropropene | 75(100); 77 (33) | 75 |
| Trichloroethylene | 95(100); 97 (66); 130 (90); 132 (85) | 130 |
| Dibromochloromethane | 129(100); 127 (78); 208 (13); 206 (10) | 127 |
| Cis-1,3-dichloropropene | 75(100); 77 (33) | 75 |
| 1,1,2-Trichloroethane | 83 (95); 85 (60); 97(100); 99 (63); 132 (9); 134(8) | 97 |
| Benzene | 78(100); 39 (13); 51 (18) | 78 |
| 2-Bromo-1-chloropropane | 77(100); 79 (33); 156 (5) | 77 |
| Bromoform | 171 (50); 173(100); 175 (50); 250 (4); 252(11) | 173 |
| 1,1,2,2-Tetrachloroethene | 129 (64); 131 (62); 164 (78); 166(100); | 164 |
| 1,1,2,2-Tetrachloroethane | 83(100); 85 (66); 131 (7); 133 (7); 166 (5); 168(6) | 168 |
| 1,4-Dichlorobutane | 55(100); 90 (30); 92 (10) | 55 |
| Toluene | 91(100); 92 (78) | 92 |
| Chlorobenzene | 112(100); 114 (33) | 112 |
| Ethylbenzene | 91(100); 106 (33) | 106 |

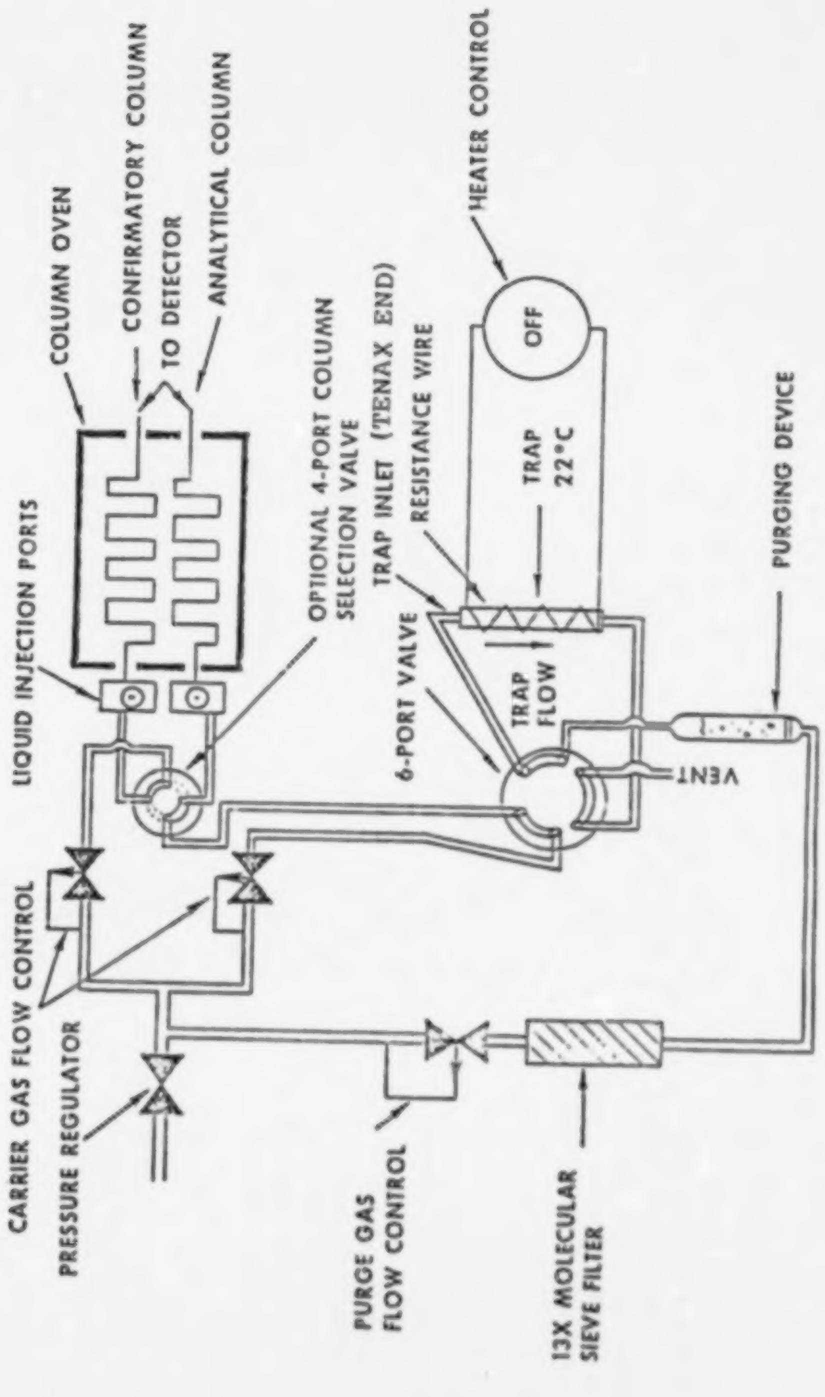

CARRIER GAS FLOW CONTROL
PRESSURE REGULATOR
LIQUID INJECTION PORTS
COLUMN OVEN
CONFIRMATORY COLUMN
TO DETECTOR
ANALYTICAL COLUMN
OPTIONAL 4-PORT COLUMN SELECTION VALVE
TRAP INLET (TENAX END)
RESISTANCE WIRE
6-PORT VALVE
TRAP FLOW
TRAP 22°C
OFF
HEATER CONTROL
VENT
PURGING DEVICE
PURGE GAS FLOW CONTROL
13X MOLECULAR SIEVE FILTER

Figure V-1. Purge-trap system (purge-sorb mode)

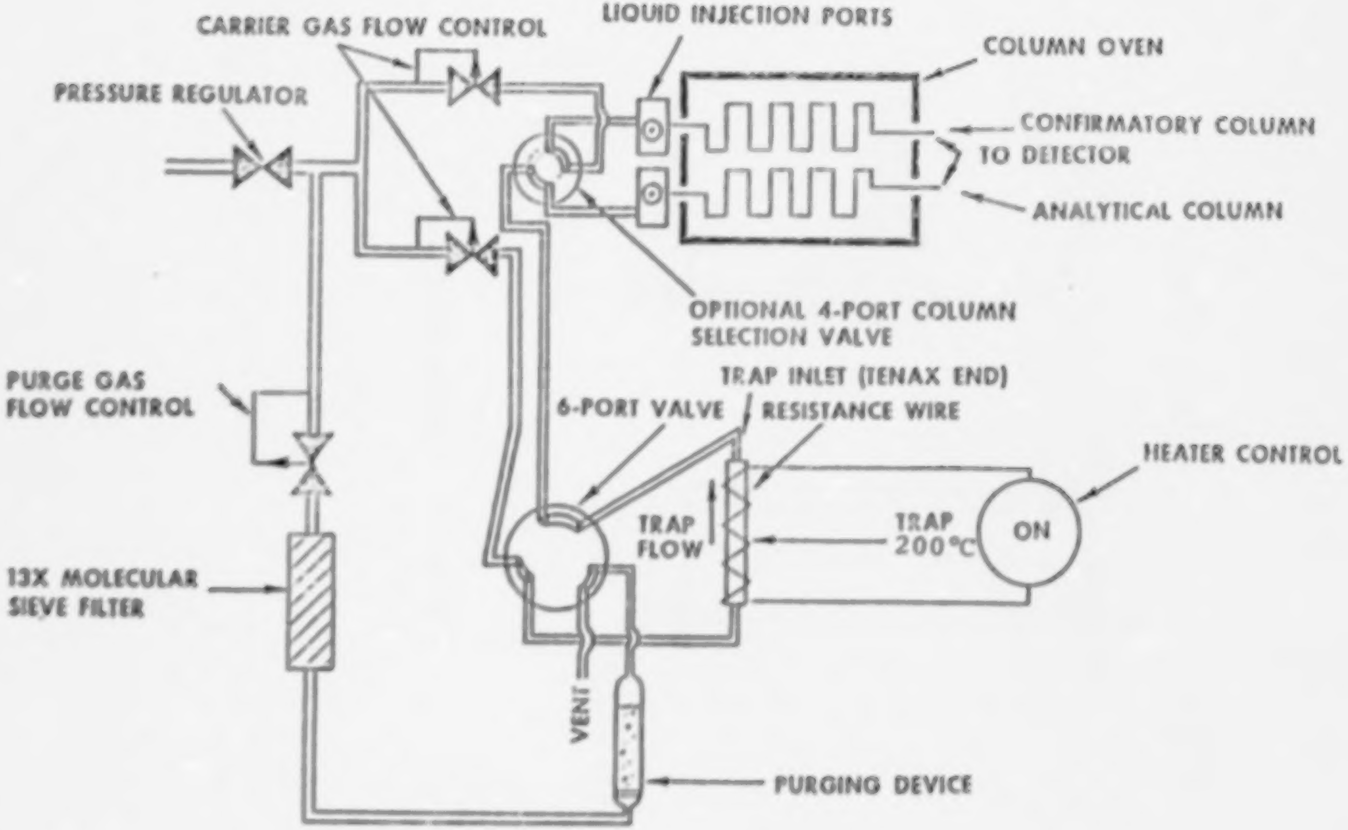

CARRIER GAS FLOW CONTROL
LIQUID INJECTION PORTS
COLUMN OVEN
PRESSURE REGULATOR
CONFIRMATORY COLUMN
TO DETECTOR
ANALYTICAL COLUMN
OPTIONAL 4-PORT COLUMN SELECTION VALVE
TRAP INLET (TENAX END)
PURGE GAS FLOW CONTROL
6-PORT VALVE
RESISTANCE WIRE
HEATER CONTROL
TRAP FLOW
TRAP 200 °C
ON
13X MOLECULAR SIEVE FILTER
VENT
PURGING DEVICE

Figure V-2. Purge-trap system (desorb mode)

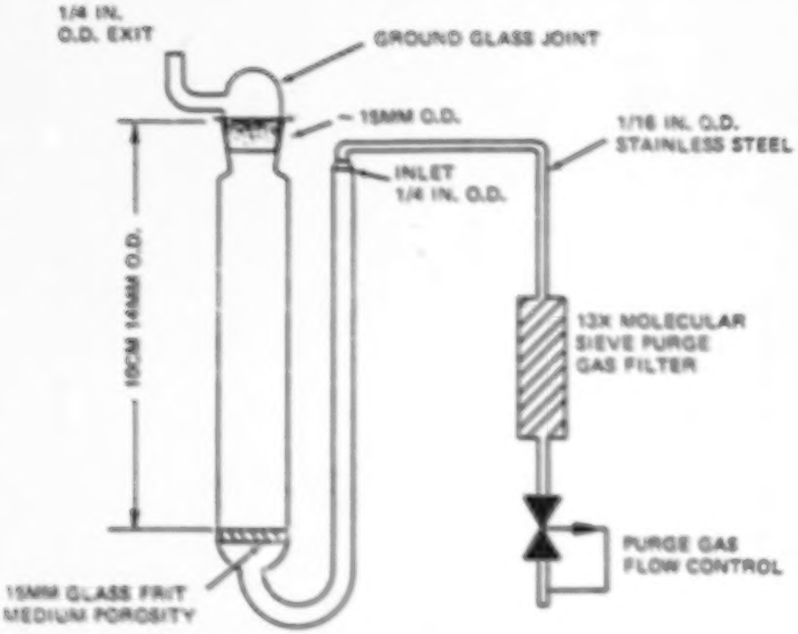

1/4 IN.
O.D. EXIT
GROUND GLASS JOINT
~ 15MM O.D.
1/16 IN. O.D.
STAINLESS STEEL
INLET
1/4 IN. O.D.
10CM 14MM O.D.
13X MOLECULAR
SIEVE PURGE
GAS FILTER
PURGE GAS
FLOW CONTROL
15MM GLASS FRIT
MEDIUM POROSITY

Figure V-3. Purging device

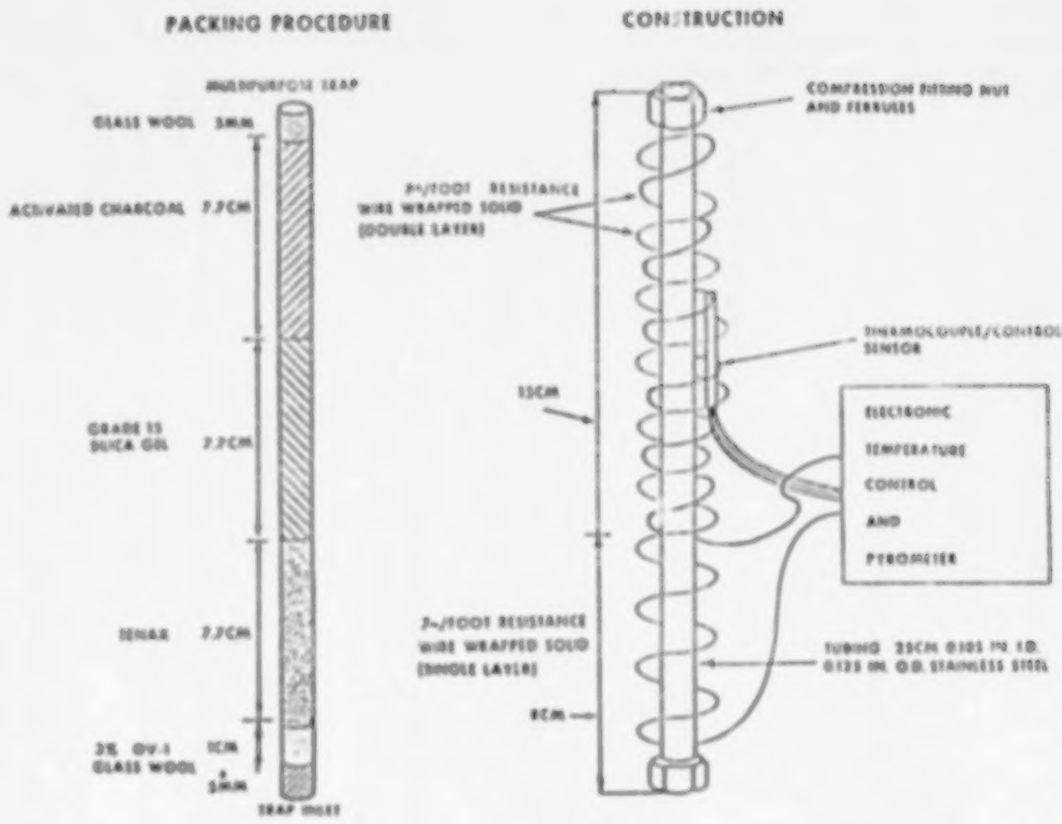

PACKING PROCEDURE
MULTIPURPOSE TRAP
GLASS WOOL 3MM
ACTIVATED CHARCOAL 7.7CM
GRADE 15 SILICA GEL 7.7CM
TENAX 7.7CM
3% OV-1 1CM
GLASS WOOL 3MM
TRAP INLET
CONSTRUCTION
COMPRESSION FITTING NUT AND FERRULES
7Ω/FOOT RESISTANCE WIRE WRAPPED SOLID (DOUBLE LAYER)
THERMOCOUPLE/CONTROLLER SENSOR
15CM
ELECTRONIC TEMPERATURE CONTROL AND PYROMETER
7Ω/FOOT RESISTANCE WIRE WRAPPED SOLID (SINGLE LAYER)
TUBING 25CM 0.105 IN. I.D. 0.125 IN. O.D. STAINLESS STEEL
8CM

Figure V-4. Sorbent trap

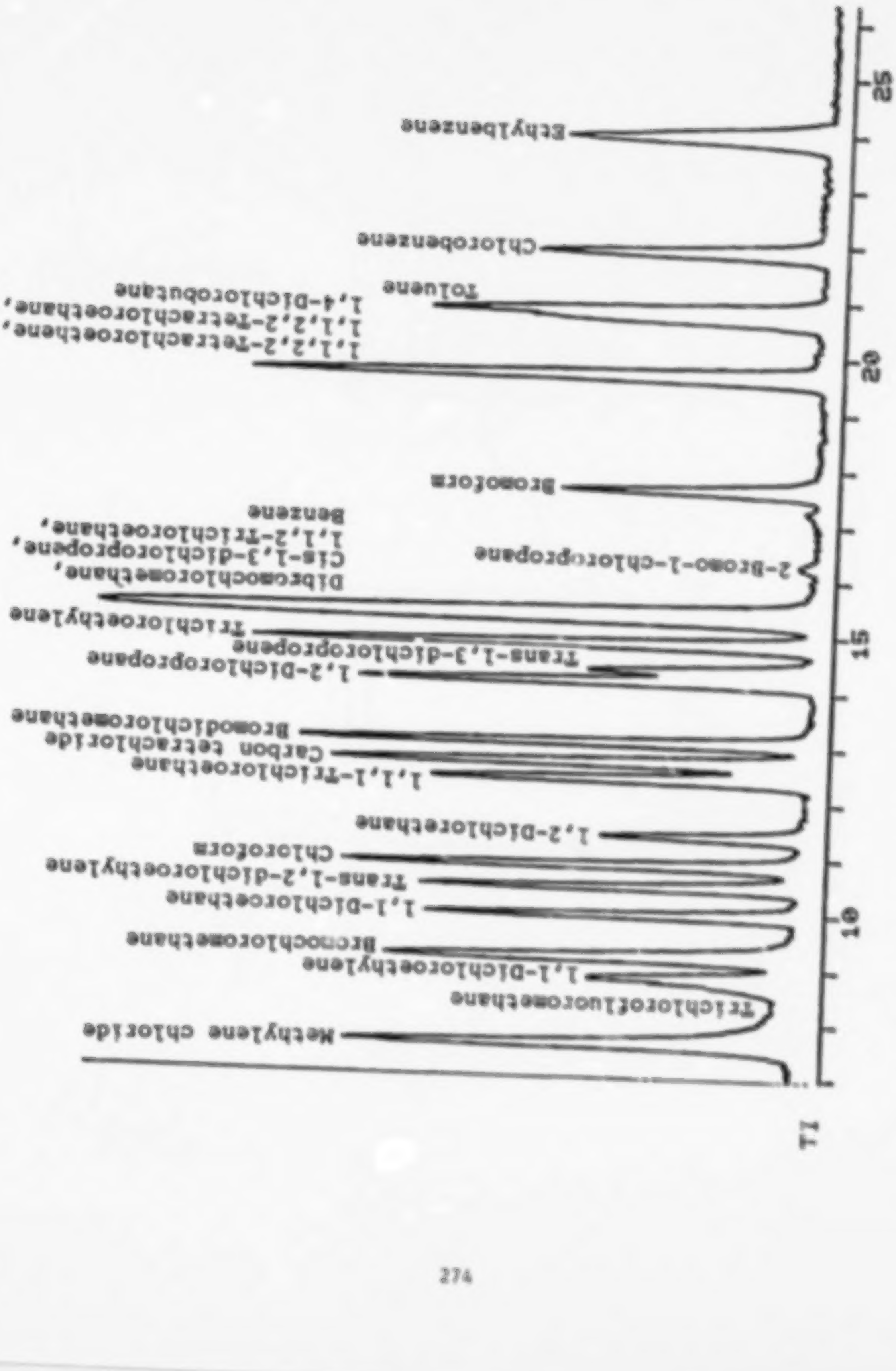


Figure V-5. Chromatogram of purgeable organics by purge and trap. GC/MS

# B. Proposed Method for Base/Neutral and Acid Extractable Semivolatile Organics in Residual Waste

# PROPOSED METHOD FOR BASE NEUTRAL AND ACID
# EXTRACTABLE SEMIVOLATILE ORGANICS
# IN RESIDUAL WASTE
(January 1981)

## 1. Scope and Application

1.1 This method covers the determination for those semivolatile organic pollutants that are solvent extractable and amenable to analysis by gas chromatography/mass spectrometry. At a minimum, the compounds listed in Table V-6 may be determined. by this method.

1.2 This method is applicable to the determination of these compounds in a variety of residual wastes. It is designed to be used to meet the survey requirements of the Office of Solid Waste (OSW). Because of the potential complexity of residual wastes, the method described below is recognized as being broad in scope; nevertheless, it was written with a priority of simplicity in mind. Of course, no single method is applicable to all waste materials and some method modification may be necessary for unusual waste types.

1.3 The detection limit of this method is usually dependent upon the level of interferences rather than instrumental limitations. The limits listed in Tables V-7 and V-8 represent levels that can be achieved in residual wastes in the absence of interferences. These levels were determined by the minimum quantity required for confirmation by the mass spectrometric method described below.

1.4 This method is recommended for use only by experienced residue analysts and persons experienced with GC/MS or under the close supervision of such qualified persons.

## 2. Summary of Method

2.1 A 40-g sample of wet residual waste is extracted with methylene chloride with the aid of a high-speed homogenizer. The sample is extracted at pH $\geq 11$ and again at pH $\leq 2$ to extract base/neutral and acidic compounds, respectively. Each extract is dried over sodium sulfate and concentrated to a volume of 5 mL or less by use of a Kuderna-Danish (K-D) evaporator. The GC/MS conditions described allow separation, identification, and quantitation of the compounds in the extracts.

2.2 The method provides a general-purpose, gel permeation chromatography (GPC) cleanup procedure to minimize interferences if they are encountered.

2.3 Identification of a compound (qualitative analysis) is performed by an analysis of the full, background-corrected mass spectrum. To identify a compound qualitatively, an Extracted Ion Current Profile (EICP) is obtained for the primary ion and at least two other ions (if available).

2.4 Quantitative analysis is performed by GC/MS with either the internal standard or the external standard technique.

2.5 A flow diagram summarizing the method is given in Figure V-6.

## 3. Interferences

3.1 Solvents, reagents, glassware, and other sample processing hardware may yield discrete artifacts causing misinterpretation. All of these materials must be demonstrated to be free from interferences under the conditions of the analysis by running method blanks. Specific selection of reagents or purification of solvents by distillation in all-glass systems may be required.

3.2 Interfering substances coextracted from the samples will vary considerably from source to source, depending upon the diversity of the residual wastes being sampled. Although general cleanup techniques are provided as part of this method, unique samples may require additional cleanup approaches to isolate the compounds of interest from interfering substances and to achieve the sensitivities stated in Tables V-7 and V-8.

3.3 Glassware must be scrupulously clean. Clean glassware as soon as possible after use by rinsing with the last solvent used. Glassware should be sealed/stored in a clean environment immediately after drying to prevent any accumulation of dust or other contaminants.

3.4 The recommended analytical procedure may not afford sufficient resolution to differentiate between certain isomeric pairs. Examples are anthracene and phenathrene, chrysene and benzo(a)-anthracene, and benzo(b)fluoranthene and benzo(k)fluoranthene. The GC retention times and mass spectral data are not sufficiently different to provide an unambiguous distinction between these compounds. Resolution may be improved with GC capillary columns. Alternative techniques should be used to identify these specific compounds.

## 4. Apparatus and Materials

4.1 Sampling equipment, for discrete or composite sampling.

4.1.1 Grab sample bottle—Amber glass, 1-liter or 1-quart volume, minimum. Bottle with a wide-mouth design is recommended. The container must be washed, rinsed with solvent, and dried before use to minimize interferences.

4.1.2 Bottle caps—Threaded to fit sample bottles. Caps must be lined with Teflon. Aluminum foil may be substituted if the sample is not corrosive.

4.1.3 Compositing equipment—Automatic or manual compositing system that incorporates glass sample containers for the collection of a minimum of 1000 mL. Sample containers must be kept refrigerated during sampling. No plastic or rubber tubing other than Teflon may be used in the system.

4.2 Extracting equipment.

4.2.1 SDT Tissumizer (Tekmar SDT 182 EN or equivalent).

4.2.2 Centrifuge (IEC CU-5000 or equivalent).

4.2.3 Screw-capped centrifuge bottles—200 mL (Scientific Products C4144) with Teflon-lined screw caps.

4.2.4 Fleakers (or beakers)—300 mL.

4.2.5 Glass syringe—50 mL equipped with a 150 mm x 5 mm ID Teflon tube.

4.3 Drying column—A 400 mm x 20 mm ID Chromaflex Column equipped with a glass wool plug (Kontes Glass K-420300 or equivalent).

4.4 Kuderna-Danish (K-D) apparatus.

4.4.1 Concentrator tube—10 mL, graduated (Kontes K-570050-1025 or equivalent). Calibration must be checked. Ground glass stopper (size 19/22 joint) is used to prevent evaporation of extracts.

4.4.2 Evaporative flask—500 mL (Kontes K-57001-0500 or equivalent). Attach to concentrator tube with springs (Kontes K-662750-0012).

4.4.3 Snyder column—Three-ball macro (Kontes K503000-0121 or equivalent).

4.4.4 Snyder column—Two-ball micro (Kontes K-569001-0219 or equivalent).

4.4.5 Teflon boiling stones (Chemplast or equivalent).

4.4.6 Tube heater/concentrator (K720001) with heating block (K720003).

4.5 Water bath for Kuderna-Danish concentrators. The bath should be used in a hood.

4.6 Gel permeation chromatography cleanup apparatus.

4.6.1 Chromatography column—500 mm x 19 mm ID (Scientific Products C-4670-106 or equivalent).

4.6.2 Bio-Beads S-X3, 200/400 mesh (Bio-Rad Laboratories 152-2750).

4.6.3 Glass wool.

4.6.4 Graduate cylinders—100 mL.

4.6.5 GPC Autoprep (Analytical Biochemistry Labs, Inc. 1002 or equivalent with 25-mm ID column containing 50 to 60 g of Bio-Beads S-X3) (optional).

4.7 Gas chromatograph—Analytical system complete with gas chromatograph capable of on-column injection. All required accessories including column supplies, gases, etc.

4.7.1 Column 1—For base/neutrals, a 6-ft glass column (1/4 in. OD x 2 mm ID) packed with 1% SP-2250 coated on 100/120 Supelcoport (or equivalent).

4.7.2 Column 2—For acids, a 6-ft glass column (1/4 in. OD x 2 mm ID) packed with 1% SP-1240 DA coated on 100/120 Supelcoport (or equivalent).

4.8 Mass spectrometer—Capable of scanning from 35 to 350 a.m.u. every 4 sec or less at 70 volts (nominal) and producing a recognizable mass spectrum (background corrected) at unit resolution from 50 ng of decafluorotriphenylphosphine (DFTPP) when the sample is introduced through the GC inlet (Reference 2). The mass spectrometer must be interfaced with a gas chromatograph equipped with an injector system designed for all-glass on-column injection on packed columns or, alternatively, equipped with a capillary injector system designed for splitless injection and capillary columns. All sections of the transfer lines must be glass or glass-lined and must be deactivated. (Use Sylon-CT, Supelco, Inc., or equivalent to deactivate.) Note: Systems utilizing a jet separator for the GC effluent are required since membrane separators may cause loss of sensitivity to small molecules, and glass frit separators may inhibit the elution of polynuclear aromatics.

4.9 A computer system must be interfaced to the mass spectrometer to allow acquisition of continuous mass scans for the duration of the chromatographic program. The computer system should also be equipped with mass storage devices for saving all data from GC/MS runs. There must be computer software available to allow searching any GC/MS run for specific ions and plotting the intensity of the ions with respect to time or scan number. The ability to integrate the area under any specific ion plot peak is essential for quantification.

## 5. Reagents

5.1 Sodium hydroxide—(ACS) 10 N in distilled water.

5.2 Hydrochloric acid—(ACS) concentrated, 12 N.

5.3 Sodium sulfate—(ACS) granular anhydrous; conditioned at 400 °C for 4 h and rinsed with methylene chloride (20 mL of solvent per gram of sodium sulfate).

5.4 Methylene chloride—Pesticide quality (Burdick and Jackson or equivalent).

5.5 Reference materials—These materials consist of assayed, authentic samples of all of the compounds to be determined and the following standards in addition: (1) internal standards, (2) surrogate standards, and (3) decafluorotriphenylphosphine (a calibration standard for the MS). Internal standards may be selected from the following list of compounds:

> Deuterated compounds. For aromatics: $d_6$-benzene, $d_8$-toluene, and $d_{10}$-anthracene. For amines: $d_5$-pyridine and $d_5$-aniline. For phenols: $d_6$-phenol. For nitroaromatics: $d_5$-nitrobenzene.

Surrogate standards are taken from this list of alternate compounds:

> Fluorinated compounds: For aromatics: pentafluorobenzene, 2-fluoronaphthalene, and 1,2,3,4,5-pentafluorobiphenyl. For phenols: pentafluorophenol, $\alpha,\alpha,\alpha$-trifluoro-m-cresol, and 2-fluorophenol.

5.6 GPC calibration solution—Prepared as a 1:1 mixture of the following:

5.6.1 Corn oil-200 mg/mL in methylene chloride.

5.6.2 Bis(2-ethylhexyl phthalate) and pentachlorophenol—4.0 mg/mL in methylene chloride.

## 6. Calibration Standards

6.1 Prepare calibration standards that contain samples of the authentic compounds to be determined, either singly or mixed together. The standards should be prepared at concentrations that will completely bracket the working range of the chromatographic system (two or more orders of magnitude are suggested). If the limit of detection of a given compound (Table V-7 or V-8) can be calculated as 20 ng injected, for example, prepare standards at 10 μg/mL, 100 μg/mL, 1000 μg/mL, etc., so that injections of 1 to 5 μL of the calibration standards will define the linearity of the detector in the working range.

6.2 Assemble the necessary gas chromatographic apparatus and establish operating parameters equivalent to those indicated in Tables V-7 and V-8. By injecting calibration standards, establish the linear range of the analytical system and demonstrate that the analytical system meets the limits of detection requirements of Tables V-7 and V-8. If the sample gives peak areas above the working range, dilute and reanalyze.

6.3 The internal standard consists of adding one or more of the selected compounds (Section 5.5) to each sample to be analyzed. This approach is acceptable for all of the extractable semi-volatile organics when internal standards meeting the following criteria can be selected:

6.3.1 No interference with other components in the sample.

6.3.2 Structural similarity to the compound to be determined.

6.3.3 The amount added approximates the concentration of the compound to be determined.

6.4 Internal standard method—The utilization of the internal standard method requires the periodic determination of response factors (RF) which are defined in Equation 1.

$$RF - (A_sC_{is})/(A_{is}C_s) \qquad (1)$$

where: $A_s$ is the integrated area of peak height of the characteristic ion for the calibration standard (an authentic sample of the compound being determined).

$A_{is}$ is the integrated area or peak height of the characteristic ion for the internal standard.

$C_{is}$ is the amount (μg) of the internal standard.

$C_s$ is the amount (μg) of the calibration standard.

The response factor (RF) must be determined over all concentration ranges of the calibration standard ($C_s$) which are being determined. Generally, the amount of the internal standard added to each extract is the same—20 or 40 μg/mL—so that $C_{is}$ remains constant. Use a minimum of three concentrations over the range of interest. Once this relative response factor has been determined, it should be verified daily by injecting at least one standard solution containing an internal standard. If a significant change has occurred, a new response factor must be established. If significant changes in individual response factors have occurred, a new calibration standard solution should be analyzed. To quantify, add the internal standard to the concentrated sample extract no more than a few minutes before injecting into the GC/MS. To minimize the possibility of losses due to evaporation, adsorption or chemical reaction, calculate the concentration by using Equation 2 in Section 17.2 with an appropriate response factor from Equation 1. (Ideally, the response factor will not change with concentration.)

6.5 The external standard method can be used at the discretion of the analyst and is recommended when the criteria for use of the internal standard cannot be met. Prepare a master calibration curve using a minimum of three standard solutions of each of the compounds that are to be measured. Plot concentrations versus integrated areas or peak heights (selected characteristic ion for GC/MS). One point on each curve should approach the limit of detection in Table V-7 or V-8. After the master instrument calibration curves have been established, they should be verified daily by injecting at least one standard solution. If significant drift has occurred, a new calibration curve must be constructed. Calculate the concentration by using the equation in Section 17.3.

## 7. Quality Control and Quality Assurance

7.1 Before processing any samples, demonstrate through the analysis of a method blank that all glassware and reagents are interference-free. Each time a set of samples is extracted or there is a change in reagents, a method blank should be processed as a safeguard against chronic laboratory contamination.

7.2 Standard quality assurance practices should be used with this method. Field replicates should be collected to determine the precision of the sampling technique. Laboratory replicates should be analyzed to determine the precision of the analysis. Fortified (spiked) samples should be analyzed to determine the accuracy of the analysis. Field blanks should be analyzed to check for contamination introduced during sampling and transportation.

7.3 GC/MS system performance evaluation is required each day the system is used for samples or reagent blanks. A sample of 50 ng of decafluorotriphenylphosphine is injected into the system, the mass spectrum is acquired, and the background-corrected spectrum is plotted. Criteria established in Reference 2 must be met. The analyst should also demonstrate that the analytical conditions employed result in sharp total ion current peaks for 100 ng of benzidine on the SP-2250 column when this column is used for base/neutrals, and 250 ng of pentachlorophenol on the SP-1240 DA column when it is used for acids. All plots from the performance evaluation must be retained as proof of valid performance (Reference 3).

7.4 Further details on quality control and quality assurance are given in the QA/QC protocol.

## 8. Sample Collection, Preservation, and Handling

8.1 Grab samples must be collected in glass containers. Conventional sampling practices should be followed (Reference 5). Composite samples should be collected in glass containers maintained at 4 °C and in accordance with the specific requirements of a given sampling and analysis problem. Automatic sampling equipment must be free of Tygon and other potential sources of contamination.

8.2 The samples must be iced or refrigerated from the time of collection until the time of extraction. Chemical preservatives should not be used in the field unless more than 24 h will elapse before delivery of samples to the laboratory. If the samples will not be extracted within 24 h of the time of collection, preservation techniques should be considered to prevent deterioration of the samples.

8.3 All samples must be extracted within 2 d of sample receipt and within 5 d of sample collection. The analyses must be completed within 7 d of collection.

## 9. Sample Extraction (Base/Neutrals)

9.1 Thoroughly mix the residual waste sample by homogenizing it in the sample bottle. Weigh several 40-g aliquots into 250-mL centrifuge bottles; spike the aliquots with the surrogate spikes according to the QA/QC protocol. Add 40 mL of deionized, distilled water. Aqueous samples may not require the addition of water.

9.2 Adjust the pH of the sample with 10 N sodium hydroxide to a pH of 11 or greater. Mix briefly with the homogenizer to ensure uniform sample pH.

9.3 Add 60 mL of methylene chloride to each centrifuge bottle and homogenize briefly. Rinse the homogenizer with a minimum volume of water and then with about 5 to 10 mL of methylene chloride. An additional amount of methylene chloride may be added until the total liquid level is near the top of the centrifuge bottle.

9.4 Centrifuge the sample aliquots at 1400 R.C.F. for 15 min. The mixture will separate into an aqueous layer over the methylene chloride extract. A solid cake or emulsion may form at the water-methylene chloride interface. If the emulsion interface between layers has more than one-half the volume of the solvent layer, the analyst may employ a smaller sample aliquot to complete the phase separation. The optimum technique will depend upon the total solid content of the sample. Withdraw the organic extract from the centrifuge bottle with a 50-mL glass syringe that has been equipped with a 150 mm x 5 mm ID Teflon tube. Discharge the extract into a 300-mL fleaker.

9.5 Add a second 60-mL volume of methylene chloride to the centrifuge bottle and complete the extraction procedure a second time, combining the extracts in the fleaker.

9.6 Perform a third extraction in the same manner. Pour the combined methylene chloride extracts through a drying column containing a 60-mm layer of anhydrous sodium sulfate and into a 500-mL K-D flask equipped with a 10-mL concentrator tube. Rinse the fleaker with 20 to 40 mL of methylene chloride. Pour the rinse solvent through the drying column. Seal the combined extracts and rinse solvent, label the base/neutral fraction, and proceed with the acid extraction.

## 10. Sample Extraction (Acids)

10.1 Adjust the pH of the water, previously extracted for base-neutrals, with hydrochloric acid to a pH of 2 or less. The acid must be added slowly and with instant mixing to minimize foaming of the sample.

10.2 Extract the sample again by procedures described in Sections 9.3 to 9.6. Discard the extracted residual wastewater aliquots. Seal the combined extracts, label as the acid fraction.

## 11. Extract Concentration (Base/Neutrals and Acids)

11.1 Add one Teflon boiling stone to the 500-mL K-D flask equipped with a 10-mL concentrator tube and attach a three-ball macro-Snyder column. Prewet the Snyder column by adding about 1 mL of methylene chloride through the top. Place the K-D apparatus on a warm water bath (60 to 65 °C) so that the concentrator tube is partially immersed in the water and the entire lower rounded surface of the flask is bathed with water vapor. Adjust the vertical position of the apparatus and the water temperature as required to complete the concentration in 15 to 20 min. At the proper rate of distillation the balls of the column actively chatter but the chambers do not flood. When the liquid has reached an apparent volume of 1 mL, or when distillation appears to cease, remove the K-D apparatus from the water bath and allow the solvent to drain for at least 10 min while cooling. Remove the Snyder column and rinse the flask and its lower joint into the concentrator tube with 1 to 2 mL of methylene chloride from a Teflon squeeze bottle. Reserve the concentrated extract for analysis or additional concentration as in Section 11.2.

11.2 If additional concentration is required, add a clean boiling stone and attach a two-ball micro-Snyder column to the concentrator tube referred to in Section 11.1. Prewet the column by adding about 0.5 mL of methylene chloride through the top. Place the K-D apparatus in the tube heater that is preset at a temperature required to complete the concentration in 5 to 10 min. At the proper rate of distillation the balls of the column actively chatter but the chambers do not flood. When the liquid reaches an apparent volume of about 0.5 mL, remove the K-D from the tube heater and allow the solvent to drain and cool for at least 10 min. Remove the micro-Snyder column and rinse its lower joint into the concentrator tube with approximately 0.2 mL of methylene chloride. Adjust the final volume to 5.0 mL. This final volume adjustment will depend on the concentrations of compounds being determined as well as the nature of the residual wastewater. Reserve the concentrated extract for analysis or cleanup as described in Section 12. Cleanup will be required on all samples containing interference.

## 12. Extract Cleanup

12.1 Place 20 to 25 g of Bio-Beads S-X3 in a 200-mL beaker. Cover the beads with methylene chloride and allow the beads to swell overnight before packing a 500 mm x 19 mm ID chromatographic column. Put a glass wool plug in the bottom of the column. Transfer the swelled beads to the column and continue to rinse the packed column with methylene chloride. Add to the top of the packed column a glass wool plug followed by a layer of glass beads that will prevent the Bio-Beads from floating to the top of the eluting solvent. Wash the column with about 200 mL of methylene chloride. Just prior to exposure of the GPC packing, stop the elution with methylene chloride by closing the stopcock on the chromatography column. Discard the eluted solvent.

12.2 Transfer 5 mL of the GPC calibration solution to the Bio-Beads S-X3 column. Drain the column into a 12-mL graduated centrifuge tube until the liquid is just above the surface of the GPC packing. Wash the calibration solution on the column with several 1-mL aliquots of methylene chloride. Elute the column with 200 mL of methylene chloride and collect 10-mL fractions. Analyze the fractions for bis(2-ethylhexyl) phthalate and pentachlorophenol by GC/FID on a 1% SP-1240 DA column. Determine the corn oil elution pattern by evaporation of each fraction to dryness followed by gravimetric determination of the residue. Plot the concentration of each component in each fraction versus the total eluent volume. Determine the range of eluent volumes following ≥85% of the corn oil, including all of the phthalate and all of the pentachlorophenol, and including 50 mL beyond the pentachlorophenol. This range of eluent volumes dictates the range in which all of the cleaned sample extract will be recovered in Section 12.3. A typical calibration may result in the following procedure: discard the first 60 mL and retain the next 110 mL for sample analysis.

12.3 Apply the above GPC separation procedure to an aliquot (1 to 4 mL) of the base/neutral or acid concentrate from Section 11.2 The volume of concentrate submitted to GPC is determined by the total material in the concentrate. Determine a residue weight of the concentrate by placing a 1-mL aliquot on a tared aluminum foil pan, allowing the solvent to evaporate, and reweighing the pan. The volume of extract submitted to GPC should not exceed the capacity of the column—approximately 200 mg.

12.4 Collect the GPC-cleaned sample extract by the procedure determined in Section 12.2, by allowing the correct range of column effluent to pass through a 60-mm layer of sodium sulfate into a 500-mL K-D flask equipped with a 10-mL concentrator tube. Rinse the drying column with 10 to 20 mL of methylene chloride into the K-D flask.

## 13. GPC-Cleaned Extract Concentration

13.1 Concentrate the GPC-cleaned extract as described in Section 11.

13.2 Transfer the cleaned, concentrated extract to a 6-mL serum Teflon-capped bottle and store at 4 °C for GC/MS analysis.

## 14. Gas Chromatography-Mass Spectrometry of Base/Neutrals Fraction

14.1 Table V-7 summarizes the recommended gas chromatographic column materials and operating conditions for the instrument. Included in this table are estimated retention times and sensitivities that can be achieved by this method for base/neutral extractable semivolatile organics. An example of the separations achieved is shown in Figure V-7.

14.2 Calibrate the system daily with a minimum of one injection of calibration standards. Insure that the GC/MS system meets the criteria in Reference 2 by injecting 50 ng of DFTPP through the GC inlet system.

14.3 Transfer the sample extract (from Section 11.1, 11.2, or 12.4) into a solvent-tight container. Do not add the internal standard at this time. The recommended container is a standard 2-mL serum vial equipped with a Teflon-lined rubber septum and crimp cap. When the sample extract is not being used for analysis, it is stored in a serum vial with an unpierced septum and in the dark below 4 °C.

14.4 Internal standard—Add 40 μg of the internal standard (20 μL of a solution having a concentration of 2 μg/μL) to 1 mL of the sample extract just prior to analysis. Mix thoroughly.

14.5 Inject 2 to 5 μL of the sample extract into the GC/MS. The solvent-flush technique is preferred. Smaller (1.0-μL) volumes can be injected if automatic devices are employed. Record the volume injected to the nearest 0.05 μL, and the resulting peak size in area units.

14.6 If the peak area exceeds the linear range of the system, dilute the extract and reanalyze.

14.7 At the beginning of each day that base/neutral analyses are to be performed, inject 100 ng of benzidine either separately or as a part of a standard mixture that may also contain 50 ng of DFTPP. The tailing factor for benzidine should be less than three. Calculation of the tailing factor is given in Reference 2 and described in Figure V-9.

## 15. Gas Chromatography/Mass Spectrometry of Acid Fraction

15.1 Table V-8 summarizes the recommended gas chromatographic column materials and operating conditions for the instrument. Included in this table are retention times and sensitivities that can be achieved by this method for acid extractable semivolatile organics. The limits of detection given in Table V-8 refer to the amounts required to obtain MS confirmation by the methods described below. Chromatography of nitrophenols is poor. An example of the separations achieved is shown in Figure V-8.

15.2 At the beginning of each day that acid fraction analyses are to be performed, inject 250 ng of pentachlorophenol either separately or as part of a standard mixture that may also contain DFTPP. The tailing factor for pentachlorophenol should be less than five. Calculation of the tailing factor is given in Reference 2 and described in Figure V-9.

15.3 After acceptable instrumental response is demonstrated, proceed with the analysis as described for the base/neutral fraction. (Sections 14.1 through 14.6).

## 16. Reduction of Data from the Mass Spectrometer

16.1 The complete background-corrected mass spectrum is compared to a reference mass spectrum, from either an authentic sample or a library spectrum, to provide qualitative identification. All of the following criteria must be met:

16.1.1 The retention time relative to that of the internal standard at the experimental mass spectrum must be within ±60 sec of the relative retention time of the authentic compound.

16.1.2 The ratios of the three EICP peak heights must agree within ±20% with the ratios of the relative intensities for these ions in a reference mass spectrum. The reference mass spectrum can be obtained from either a standard analyzed through the GC/MS system or a reference library.

16.1.3 Structural isomers that have very similar mass spectra (phenanthrene and anthracene; benzo(a)pyrene and benzo(e)-pyrene, etc.) can be explicitly identified if the resolution between the isomers in a standard mixture is acceptable. Acceptable resolution is achieved if the valley height between isomers is less than 25% of the sum of the two peak heights. Otherwise, structural isomers are identified as isomeric pairs.

16.2 In samples that contain an inordinate number of interferences, the chemical ionization (CI) mass spectrum may make identification easier. The use of chemical ionization MS to support EI is encouraged but not required.

## 17. Calculations (Base/Neutrals and Acids)

17.1 When a compound has been identified, the quantification of that compound will be based on the integrated area from the specific ion plot of the first-listed characteristic ion in Table V-9 or V-10. If the sample produces an interference for the first-listed ion, use a secondary ion to quantify. Quantification will be done by the internal or external standard method.

17.2 Internal standard method—By addition of a constant known amount of internal standard ($C_{is}$ in micrograms) to every sample extract, the concentration of a pollutant ($C_o$) in micrograms per gram of the sample is calculated by use of Equation 2.

$$C_o = \frac{(A_s)\ (C_{is})}{(A_{is})\ (RF)(W_o)} \cdot DF \qquad (2)$$

where: $W_o$ is the weight of the original sample in grams.

DF is the dilution factor.

Other terms are defined in Section 6.4.

17.3 External standard method—The concentration of a pollutant in the sample is determined by use of Equation 3.

$$C_o = \frac{(A)\ (V_t)}{(V_i)\ (W_s)} \qquad (3)$$

where: A = mass of compound from calibration curve (μg).

$V_i$ = volume of extracted injected (μL).

$V_t$ = volume of total extract (μL).

$W_s$ = weight of sample extracted (g).

17.4 Report all final results to at least two significant figures. Report results in micrograms per gram without correction for recovery data. When duplicate and spiked samples are analyzed, all data obtained should be reported. Recovery data, relative response ratios, and response factors from MS analysis should be included in the data.

17.5 In order to minimize unnecessary GC/MS analysis of method blanks and field blanks, the field blank may be run on a GC/FID equipped with an appropriate SP-2250 or SP-1240 DA column. If no peaks are seen of intensities equal to or greater than that of the internal standard, then it is not necessary to do a GC/MS analysis of the blank. If such peaks are seen, then the field blank must be submitted for complete analysis.

## 18. References

18.1 Method 610, Polynuclear Aromatic Hydrocarbons, U.S. EPA, EMSL, Cincinnati, Ohio 45268, 1979.

18.2 "Reference Compound to Calibrate Ion Abundance Measurement in Gas Chromatography—Mass Spectrometry Systems," J. W. Eighelberger, L. E. Harris, and W. L. Budde, *Anal. Chem.* *47*, 995-1000, 1975.

18.3 "Quality Assurance and Quality Control Procedures for Screening and Verification of Industrial Effluents for Priority Pollutants," U. S. EPA, EMSL, Cincinnati, Ohio 45268, 1979.

18.4 "Development of Analytical Test Procedures for the Measurement of Organic Priority Pollutants in Sludges and Sediments," MRI Project No. 4583-A, EMSL, Cincinnati, Ohio 45268, 1979.

18.5 "Samplers and Sampling Procedures for Hazardous Waste Streams," EPA-600/2-80-018, January, 1980.

18.6 "Development of Analytical Test Procedures for the Measurement of Organic Priority Pollutants in Sludge and Sediments," Midwest Research Institute, Final Report EPA Contract No. 68-03-2695, June 26, 1979.

## TABLE V-6. BASE/NEUTRALS AND ACID EXTRACTABLES DETERMINED BY THE PROPOSED METHOD

| Base/Neutrals |
|---|
| 1,3-Dichlorobenzene |
| 1,4-Dichlorobenzene |
| 1,2-Dichlorobenzene |
| Hexachloroethane |
| Bis(2-chloroethyl) ether |
| Bis(2-chloroisoporpyl) ether |
| Hexachlorobutadiene |
| Nitrobenzene |
| Naphthalene |
| 1,2,4-Trichlorobenzene |
| Bis(2-chloroethoxy)methane |
| N-nitrosodi-n-propylamine |
| Hexachlorocyclopentadiene |
| 2-Chloronaphthalene |
| Isophorone |
| Acenaphthylene |
| Acenaphthene |
| Dimethyl phthalate |
| 2,6-Dinitrotoluene |
| Fluorene |
| 2,4-Dinitrotoluene |
| 1,2-Diphenylhydrazine |
| 4-Chlorophenyl phenyl ether |
| Diethyl phthalate |
| N-nitrosodiphenylamine |
| Hexachlorobenzene |
| 4-Bromophenyl phenyl ether |
| Phenanthrene/anthracene |
| Di-n-butyl phthalate |
| Fluoranthene |
| Pyrene |
| Benzidine |
| Butylbenzyl phthalate |
| Bis(2-ethylhexyl) phthalate |
| Chrysene/benzo(a)anthracene |
| 3,3'-Dichlorobenzidine |
| Di-n-octyl phthalate |
| Benzo(b)fluoroanthrene/ benzo(k)fluoranthene |
| Benzo(a)pyrene |
| Indeno(1,2,3-cd)pyrene |
| Dibenzo(ah)anthracene |
| Benzo(ghi)perylene |

(Continued)

TABLE V-6. BASE/NEUTRALS AND ACID EXTRACTABLES DETERMINED BY THE PROPOSED METHOD (CONTINUED)

| Acids |
|---|
| 2-Chlorophenol |
| 2-Nitrophenol |
| Phenol |
| 2,4-Dimethylphenol |
| 2,4-Dichlorophenol |
| 2,4,6-Trichlorophenol |
| 4-Chloro-3-methylphenol |
| 2,4-Dinitrophenol |
| 4,6-Dinitro-o-cresol |
| Pentachlorophenol |
| 4-Nitrophenol |

## TABLE V-7. ELUTION ORDER AND DETECTABILITIES OF BASE/NEUTRAL EXTRACTABLES BY THE GC/MS METHOD[a]

| Compound | Relative retention time[b] | Limit of detection[c] | |
|---|---|---|---|
| | | ng injected | μg/g of sample |
| 1,3-Dichlorobenzene | 0.19 | 20 | 0.3 |
| 1,4-Dichlorobenzene | 0.21 | 20 | 0.3 |
| 1,2-Dichlorobenzene | 0.24 | 20 | 0.3 |
| Hexachloroethane | 0.24 | 20 | 0.3 |
| Bis(2-chloroethyl) ether | 0.35 | 20 | 0.3 |
| Bis(2-chloroisopropyl) ether | 0.35 | 20 | 0.3 |
| Hexachlorobutadiene | 0.44 | 20 | 0.3 |
| Nitrobenzene | 0.46 | 20 | 0.3 |
| Naphthalene | 0.47 | 20 | 0.3 |
| 1,2,4-Trichlorobenzene | 0.49 | 20 | 0.3 |
| Bis(2-chloroethoxy)methane | 0.53 | 20 | 0.3 |
| N-nitrosodi-n-propylamine | 0.58 | 20 | 0.3 |
| Hexachlorocyclopentadiene | 0.58 | 20 | 0.3 |
| 2-Chloronaphthalene | 0.66 | 20 | 0.3 |
| Isophorone | 0.68 | 20 | 0.3 |
| Acenaphthylene | 0.73 | 20 | 0.3 |
| Acenaphthene | 0.76 | 20 | 0.3 |
| Dimethyl phthalate | 0.83 | 20 | 0.3 |
| 2,6-Dinitrotoluene | 0.83 | 20 | 0.3 |
| Fluorene | 0.83 | 20 | 0.3 |
| 2,4-Dinitrotoluene | 0.87 | 20 | 0.3 |
| 1,2-Diphenylhydrazine* | 0.87 | 20 | 0.3 |
| 4-Chlorophenyl phenyl ether | 0.88 | 20 | 0.3 |
| Diethyl phthalate | 0.89 | 20 | 0.3 |
| N-nitrosodiphenylamine** | 0.89 | 20 | 0.3 |
| Hexachlorobenzene | 0.90 | 20 | 0.3 |
| 4-Bromophenyl phenyl ether | 0.95 | 20 | 0.3 |
| Phenanthrene/anthracene | 1.00 | 20 | 0.3 |
| Di-n-butyl phthalate | 1.12 | 20 | 0.3 |
| Fluoranthene | 1.19 | 20 | 0.3 |
| Pyrene | 1.22 | 20 | 0.3 |
| Benzidine | 1.36 | 20 | 0.3 |
| Butylbenzyl phthalate | 1.37 | 20 | 0.3 |
| Bis(2-ethylhexyl) phthalate | 1.41 | 20 | 0.3 |
| Chrysene/benzo(a)anthracene | 1.42 | 20 | 0.3 |
| 3,3'-Dichlorobenzidine | 1.49 | 20 | 0.3 |

(Continued)

TABLE V-7. ELUTION ORDER AND DETECTABILITIES OF BASE/NEUTRAL EXTRACTABLES BY THE GC/MS METHOD[a] (CONTINUED)

| Compound | Relative Retention time[b] | Limit of detection[c] | |
|---|---|---|---|
| | | ng injected | μg/g of sample |
| Di-n-octyl phthalate | 1.51 | 20 | 0.3 |
| Benzo(b)fluoranthene/ benzo(k)fluoranthene | 1.58 | 20 | 0.3 |
| Benzo(a)pyrene | 1.58 | 20 | 0.3 |
| Indeno(1,2,3-cd)pyrene | 1.86 | 50 | 0.8 |
| Dibenzo(ah)anthracene | 1.87 | 50 | 0.8 |
| Benzo(ghi)perylene | 1.94 | 50 | 0.8 |

* Detected as azobenzene.

** Detected as diphenylamine.

a. Six-foot glass column (1/4 in. OD x 2 mm ID) packed with 1% SP-2250 coated on 100/120 mesh Supelcoport. Carrier gas: nitrogen at 30 mL/min. Temperature program: isothermal for 4 min at 50 °C, then 8° per minute to 270 °C. Hold at 270 °C for 15 min. If desired, capillary columns may be used.

b. Relative to $d_{10}$-anthracene (internal standard) at 19.7 min.

c. This is a minimum level at which the entire analytical system must give recognizable mass spectra (background corrected) and acceptable calibration points. The overall level of detection is based on a 2-μL injection of the extract from a 40-g sample of residual waste that has been extracted and concentrated to a volume of 2.0 mL.

## TABLE V-8. ELUTION ORDER AND DETECTABILITIES OF ACID EXTRACTABLES BY THE GC/MS METHOD[a]

| Compound | Relative retention time[b] | Limit of detection[c] ng injected | Limit of detection[c] µg/g of sample |
|---|---|---|---|
| 2-Chlorophenol | 0.70 | 50 | 0.8 |
| 2-Nitrophenol | 0.79 | 50 | 0.8 |
| Phenol | 1.00 | 50 | 0.8 |
| 2,4-Dimethylphenol | 1.19 | 50 | 0.8 |
| 2,4-Dichlorophenol | 1.24 | 50 | 0.8 |
| 2,4,6-Trichlorophenol | 1.54 | 50 | 0.8 |
| 4-Chloro-3-methylphenol | 1.73 | 50 | 0.8 |
| 2,4-Dinitrophenol | 2.11 | 200 | 3.1 |
| 2,6-Dinitro-o-cresol | 2.14 | 200 | 3.1 |
| Pentachlorophenol | 2.44 | 50 | 0.8 |
| 4-Nitrophenol | 2.86 | 50 | 0.8 |

a. Six-foot glass column (1/4 in. OD x 2 mm ID) packed with 1% SP-1240 DA coated on 100/120 mesh Supelcoport. Carrier gas: nitrogen at 30 mL/min. Temperature program: isothermal for 2 min at 70 °C, then 8° per minute to 190 °C. Hold at 190 °C for 8 min. If desired, capillary column may be used.

b. Relative to $d_6$-phenol (internal standard) at 7.0 min.

c. This is a minimum level at which the entire analytical system must give recognizable mass spectra (background corrected) and acceptable calibration points. The overall level of detection is based on a 2-µL injection of the extract from a 40-g sample of residual waste that has been extracted and concentrated to a volume of 2.0 mL.

TABLE V-9. CHARACTERISTIC IONS OF BASE/NEUTRAL EXTRACTABLES

| Compound | EI ions (relative intensity) | Ion used to quantify |
|---|---|---|
| 1,3-Dichlorobenzene | 146(100), 148(64), 113(12) | 146 |
| 1,4-Dichlorobenzene | 146(100), 148(64), 113(11) | 146 |
| 1,2-Dichlorobenzene | 146(100), 148(64), 113(11) | 146 |
| Hexachloroethane | 117(100), 199(61), 201(99) | 117 |
| *Bis*(2-chloroethyl) ether | 93(100), 63(99), 95(31) | 93 |
| *Bis*(2-chloroisopropyl) ether | 45(100), 77(19), 79(12) | 45 |
| Hexachlorobutadiene | 225(100), 223(63), 227(65) | 225 |
| Nitrobenzene | 77(100), 123(50), 65(15) | 77 |
| Naphthalene | 128(100), 127(10), 129(11) | 128 |
| 1,2,4-Trichlorobenzene | 74(100), 109(80), 145(52) | 180 |
| Bis(2-chloroethoxy)methane | 93(100), 95(32), 123(21) | 93 |
| N-nitrosodi-n-propylamine | 130 (22), 42(64), 101(12) | 130 |
| Hexachlorocyclopentadiene | 237(100), 235(63), 272(12) | 237 |
| 2-Chloronaphthalene | 162(100), 164(32), 127(31) | 162 |
| Isophorone | 82(100), 95(14), 138(18) | 82 |
| Acenaphthylene | 152(100), 153(16), 151(17) | 152 |
| Acenaphthene | 154(100), 153(95), 152(53) | 154 |
| Dimethyl phthalate | 163(100), 164(10), 194(11) | 163 |
| 2,6-Dinitrotoluene | 165(100), 63(72), 121(23) | 165 |
| Fluorene | 166(100), 165(80), 167(14) | 166 |
| 2,4-Dinitrotoluene | 165(100), 63(72), 121(23) | 165 |
| 1,2-Diphenylhdyrazine | 77(100), 93(58), 105(28) | 77 |
| 4-Chlorophenyl phenyl ether | 204(100), 206(34), 141(29) | 204 |
| Diethyl phthalate | 149(100), 178(25), 150(10) | 149 |
| N-nitrosodiphenylamine | 169(100), 168(71), 167(50) | 169 |
| Hexachlorobenzene | 284(100), 142(30), 249(24) | 282 |
| 4-Bromophenyl phenyl ether | 248(100), 250(99), 141(45) | 248 |
| Phenanthrene/anthracene | 178(100), 179(16), 176(15) | 178 |
| Di-n-butyl phthalate | 149(100), 150(27), 104(10) | 149 |
| Fluoranthene | 202(100), 101(23), 100(14) | 202 |
| Pyrene | 202(100), 101(26), 100(17) | 202 |
| Benzidine | 184(100), 92(24), 185(13) | 184 |
| Butylbenzyl phthalate | 149(100), 91(50) | 149 |
| *Bis*(2-ethylhexyl) phthalate | 149(100), 167(31), 279(26) | 149 |
| Chrysene/benzo(a)anthracene | 228(100), 229(19), 226(19) | 228 |
| 3,3'-Dichlorobenzidine | 252(100), 254(66), 126(16) | 252 |
| Di-n-octyl phthalate | 149(100), 167(41) | 149 |
| Benzo(b)fluoranthene/ benzo(k)fluoranthene | 252(100), 253(23), 125(16) | 252 |
| Benzo(a)pyrene | 252(100), 253(23), 125(16) | 252 |
| Indeno(1,2,3-cd)pyrene | 276(100), 138(37), 277(25) | 276 |
| Dibenzo(ah)anthracene | 278(100), 139(24), 279(24) | 278 |
| Benzo(ghi)perylene | 276(100), 138(28), 277(27) | 276 |

TABLE V-10. CHARACTERISTIC IONS OF ACID EXTRACTABLES

| Compound | EI ions (relative intensity) | Ion used to quantify |
|---|---|---|
| 2-Chlorophenol | 128(100), 64(54), 130(31) | 128 |
| 2-Nitrophenol | 139(100), 65(35), 109 (8) | 139 |
| Phenol | 94(100), 65(17), 66(19) | 94 |
| 2,4-Dimethylphenol | 122(100), 107(90), 121(55) | 122 |
| 2,4-Dichlorophenol | 162(100), 164(58), 98(61) | 164 |
| 2,4,6-Trichlorophenol | 196(100), 198(92), 200(26) | 196 |
| 4-Chloro-3-methylphenol | 142(100), 107(80), 144(32) | 142 |
| 2,4-Dinitrophenol | 184(100), 63(59), 154(53) | 184 |
| 4,6-Dinitro-o-cresol | 198(100), 182(35), 77(28) | 198 |
| Pentachlorophenol | 266(100), 264(62), 268(63) | 266 |
| 4-Nitrophenol | 65(100), 139(45), 109(72) | 139 |

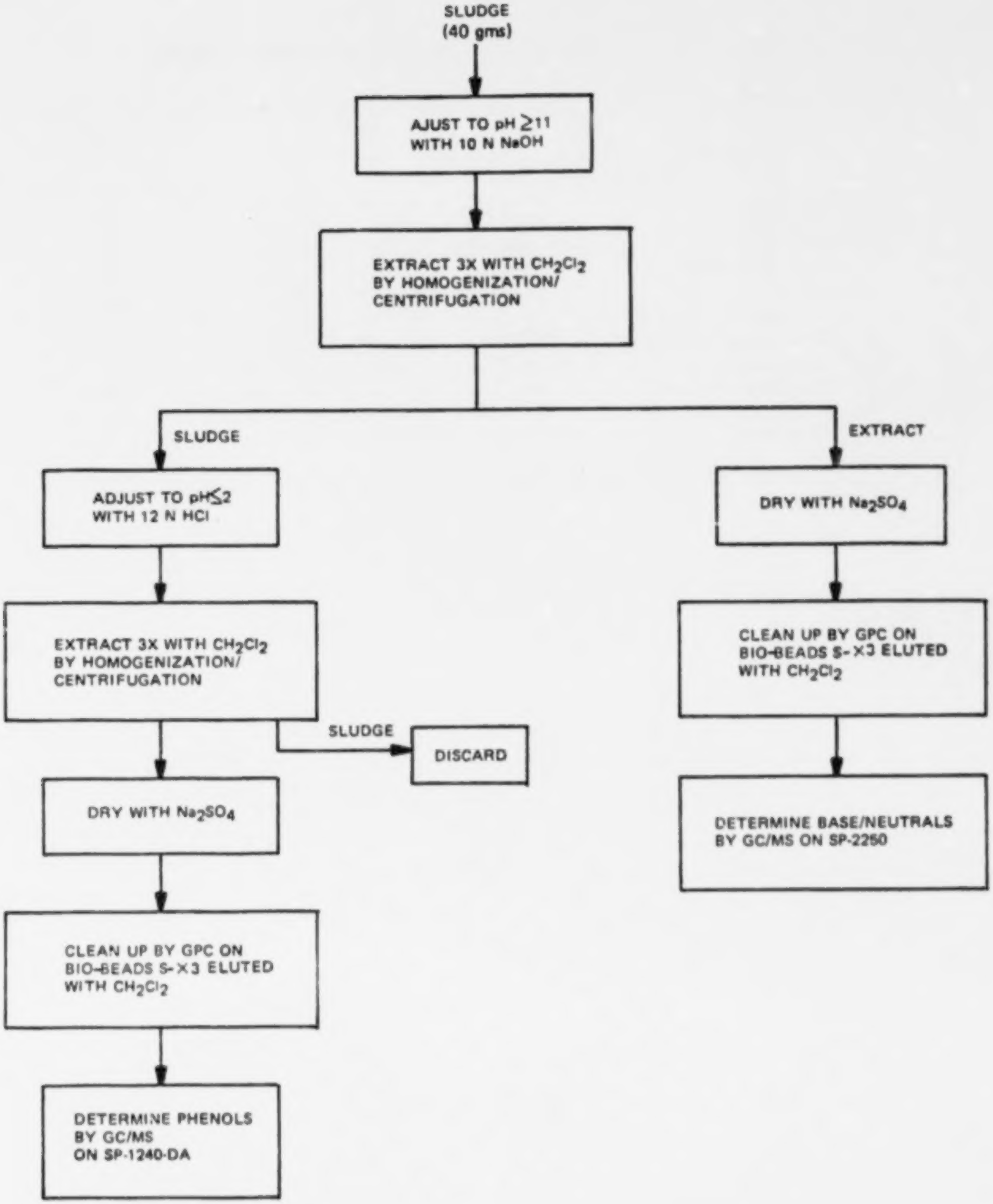

SLUDGE
(40 gms)
AJUST TO pH ≥11
WITH 10 N NaOH
EXTRACT 3X WITH $CH_2Cl_2$
BY HOMOGENIZATION/
CENTRIFUGATION
SLUDGE
EXTRACT
ADJUST TO pH≤2
WITH 12 N HCl
DRY WITH $Na_2SO_4$
EXTRACT 3X WITH $CH_2Cl_2$
BY HOMOGENIZATION/
CENTRIFUGATION
CLEAN UP BY GPC ON
BIO-BEADS S-X3 ELUTED
WITH $CH_2Cl_2$
SLUDGE
DISCARD
DRY WITH $Na_2SO_4$
DETERMINE BASE/NEUTRALS
BY GC/MS ON SP-2250
CLEAN UP BY GPC ON
BIO-BEADS S-X3 ELUTED
WITH $CH_2Cl_2$
DETERMINE PHENOLS
BY GC/MS
ON SP-1240-DA

Figure V-6. Summary of the proposed method for extractable base/neutral and acid organic compounds in residual waste

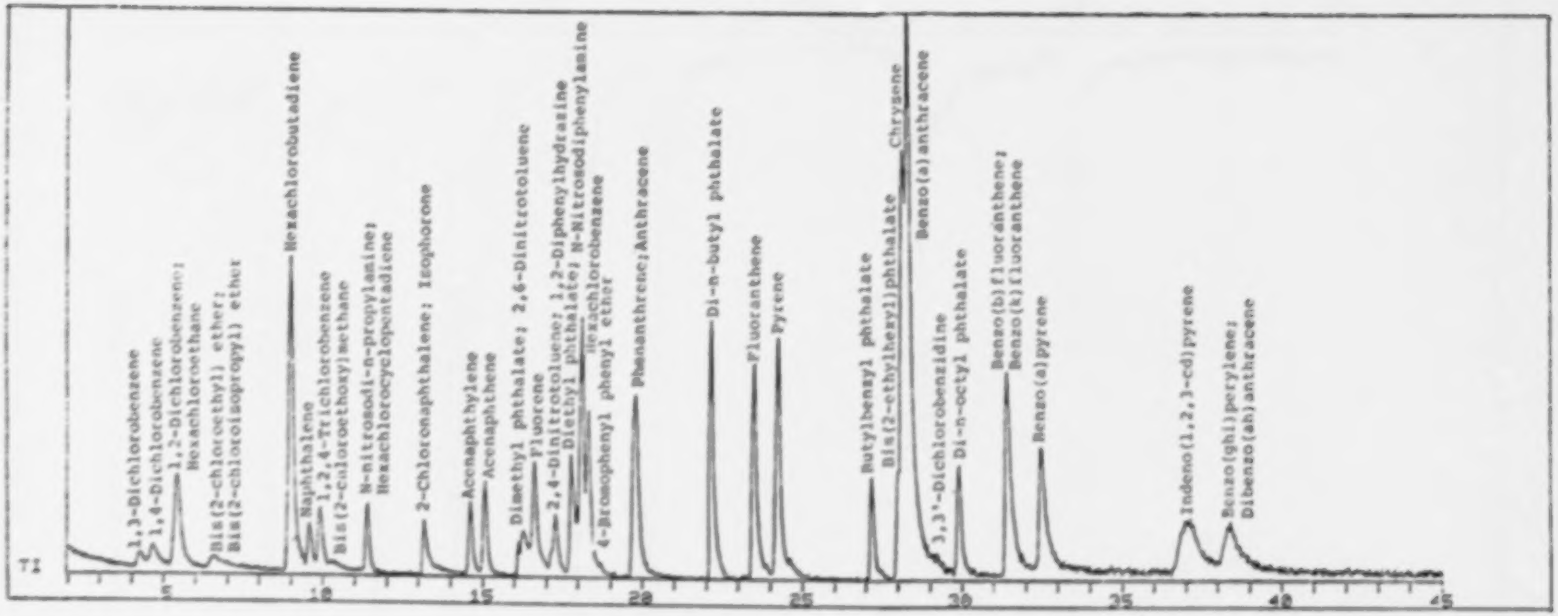


Figure V-7. Chromatogram of extractable base/neutral semivolatile organics, GC/MS

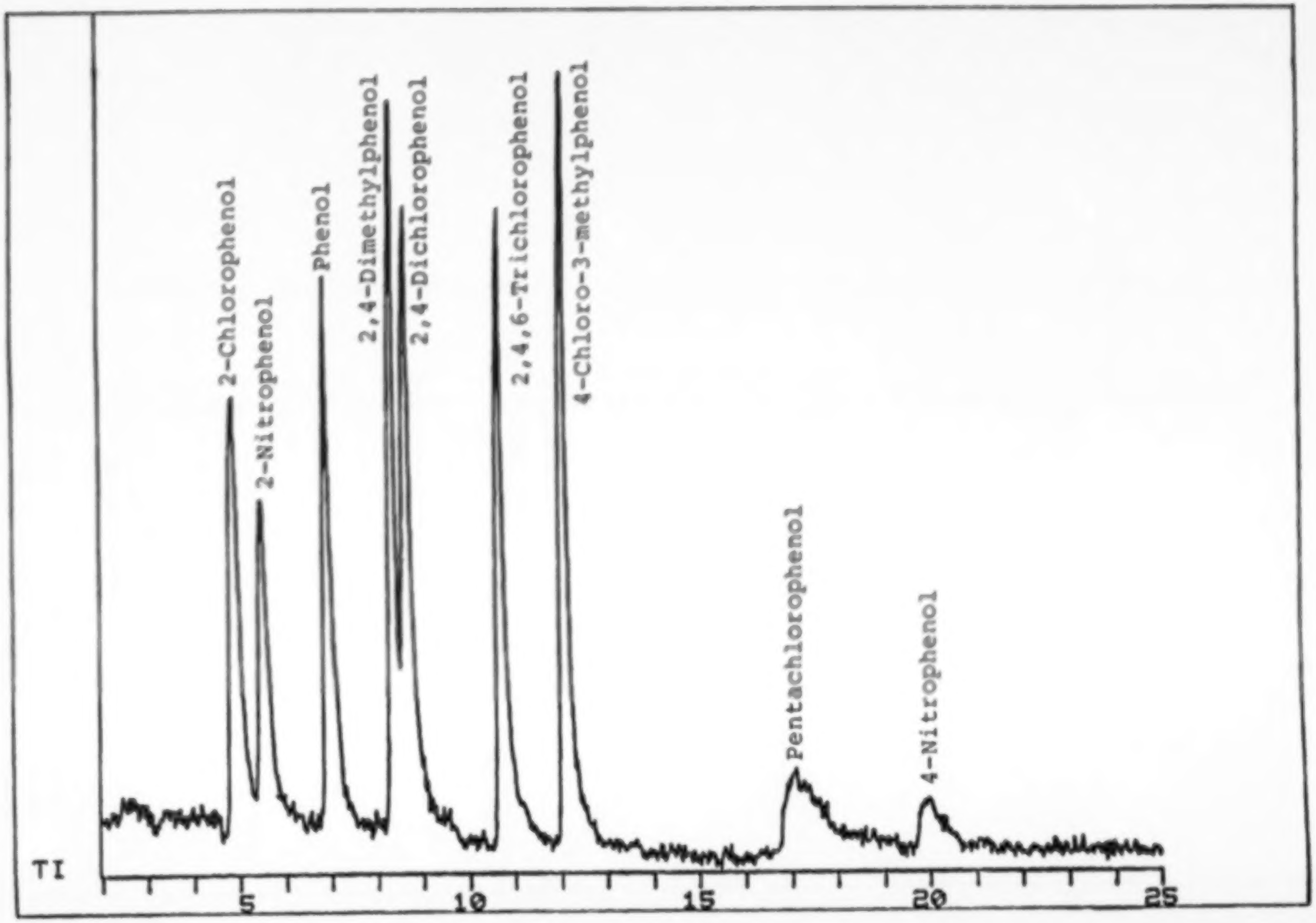
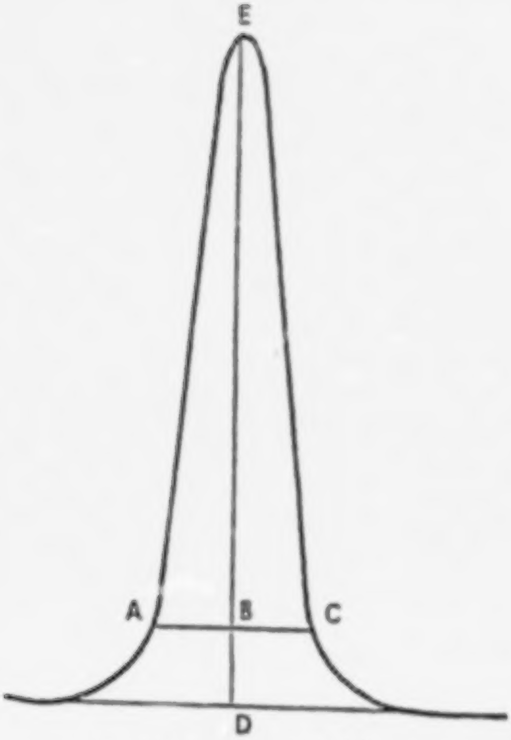


Figure V-8. Chromatogram of extractable acid semivolatile organics, GC/MS



TAILING FACTOR = $\frac{BC}{AB}$

Example calculation: Peak Height = DE = 100 mm
10% Peak Height = BD = 10 mm
Peak Width at 10% Peak Height = AC = 23 mm
AB = 11 mm
BC = 12 mm

Therefore: Tailing Factor = $\frac{12}{11}$ = 1.1

Figure V-9. Tailing factor calculation

## C. Proposed Method for the Determination of of Metals in Residual Waste

# PROPOSED METHOD FOR THE DETERMINATION
# OF METALS IN RESIDUAL WASTE
(January 1981)

## 1. Scope and Application

1.1 This method may be used for the determination of metals in residual wastes. Although the method should be applicable to a wide range of industrial wastes, it is recognized that low recoveries of certain elements may be experienced with some types of waste materials and that modifications in the method may be required in order to obtain satisfactory results.

1.2 Total elemental concentrations are determined following a digestive procedure. Since significant concentrations of elements other than the elements of interest may also be dissolved by the digestive step, each particular matrix must be examined for potential interference problems.

1.3 Detection limits, sensitivity, optimum ranges, and recommended wavelengths for the determination of selected metals by direct aspiration, furnace, gaseous hydride, and cold vapor (mercury) procedures may be obtained by consulting the instruction manual for the particular instrument being used. Sensitivity values may vary slightly with various makes and models of atomic absorption spectrometers. Actual working detection limits, however, are dependent on the sample matrix.

## 2. Summary of Method

2.1 This method describes a procedure for the determination of elements in residual wastes, after acid digestion, by atomic absorption spectroscopy.* The method of analysis may be by direct aspiration, furnace, or gaseous hydride technique, dependent on the concentration of the metal in a particular matrix. Mercury should be determined by the cold vapor procedure. A series of measurements must be performed to evaluate potential spectral, physical, and chemical interference problems as outlined in Section 4.1. Background correction should be employed for all analyses.

## 3. Definitions

3.1 Total metals—The concentration determined on an unfiltered sample following acid digestion.

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* An ICP spectrometer, if available, may also be used.

3.2 Detection limit—The concentration equivalent to a signal due to the analyte that is equal to three times the standard deviation of a series of ten replicate measurements of a reagent blank signal at the same wavelength. In an actual sample, however, the detection limit may be influenced by the sample matrix.

3.3 Sensitivity—The concentration of an element which will produce a signal of 0.0044 absorbance units.

3.4 Instrument check standard—A standard of known concentrations prepared by the analyst. It should be included in the analytical scheme with a frequency of 10%.

3.5 Reference standard—A solution obtained from an outside source having known, verified values. It must be used initially to verify the calibration standards and analyzed thereafter as a blind sample on a weekly basis.

3.6 Calibration standards—A series of known standard solutions used by the analyst for calibration of the instrument (i.e., preparation of the analytical curve).

3.7 Linear dynamic range—The concentration range over which the analytical curve remains linear.

3.8 Reagent blank—A volume of deionized, distilled water containing the same acid matrix as the calibration standards which is carried through the entire analytical scheme.

3.9 Calibration blank—A volume of deionized, distilled water acidified with nitric acid.

3.10 Method of standard addition—The standard addition technique involves the use of the unknown and the unknown plus a known amount of standard.

## 4. Interferences

4.1 Several types of interference effects may contribute to inaccuracies in the determination of trace elements. They can be summarized as follows:

4.1.1 Spectral interferences can be categorized as (1) overlap of a spectral line with that from another element; (2) unresolved overlap of molecular band spectra; and (3) background contribution from continuous or recombination phenomena. The first of these effects can be compensated for by utilizing a computer correction of the raw data, requiring measurement of the interfering element. The second effect may require selection of an alternate wavelength. The third can usually be compensated for by a background correction adjacent to the analyte line.

4.1.2 Physical interferences are generally considered to be effects associated with the sample nebulization and transport processes. Such properties as change in viscosity and surface tension can cause significant inaccuracies especially in samples that may contain high dissolved solids and/or acid concentrations. If these types of interferences are operative, they must be reduced by dilution of the sample and/or utilization of standard addition techniques.

4.1.3 Chemical interferences are characterized by molecular compound formation, ionization effects and solute vaporization effects. These, if observed, can be minimized by careful selection of operating conditions, by buffering of the sample, by matrix matching, and by standard addition procedures. These types of interferences can be highly dependent on the matrix type and the specific analyte element. Consult the manufacturer's operating manual for specific details regarding procedures to minimize chemical interferences.

4.2 It is recommended that whenever a new or unusual sample matrix is encountered, a series of tests be performed prior to reporting concentration data for analyte elements. These tests, as outlined in Sections 4.2.1 through 4.2.4, will assure the analyst that neither positive nor negative interference effects are operative on any of the analyte elements thereby distorting the accuracy of the reported values.

4.2.1 Serial dilution—If the analyte concentration is sufficiently high (minimally a factor of 10 above the instrumental detection limit after dilution), an analysis of a diluted sample should agree within 5% of the original determination (or within some acceptable control limit that has been established for that matrix). If not, a chemical or physical interference effect should be suspected.

4.2.2 Spike addition—The recovery of a spike added at a minimum level of 10X should be within 90 to 110% or within the established control limit for the matrix being analyzed. If not, a matrix effect should be suspected. The use of a standard addition analysis procedure can usually compensate for this effect.

4.2.3 Comparison with an alternate method of analysis—When a new sample matrix is investigated, comparison tests may be performed with other analytical techniques such as ICP or other approved methodology. (Caution: The standard addition technique does not detect coincident spectral overlap. If suspected, use of an alternate wavelength or comparison with an alternate method is recommended.)

4.2.4 Wavelength scanning of the analyte line region—If the appropriate equipment is available, wavelength scanning can be performed to detect potential spectral interferences.

## 5. Apparatus

5.1 Atomic absorption spectrometer.

5.1.1 Background corrector.

5.1.2 Furnace attachment.

5.1.3 Recorder, multi-range, 0-10 mV.

5.1.4 Argon gas supply, welding grade or better.

5.1.5 Acetylene gas supply, welding grade.

5.1.6 Nitrous oxide gas supply, C.P. grade.

5.1.7 Nitrogen gas supply, dry.

5.1.8 Hydrogen gas supply, C.P. grade.

5.1.9 Air supply: dry, oil-free, dust-free air from cylinders or compressed air lines.

5.2 Operating conditions—Because of the differences between various makes and models of satisfactory instruments, no detailed operating instructions can be provided. Instead, the analyst should follow the instructions provided by the manufacturer of the particular instrument being used. Sensitivity, instrumental detection limit, precision, linear dynamic range, and interference effects must be investigated and established for each individual analyte line on that particular instrument.

## 6. Reagents and Standards

6.1 The nitric acid used in the preparation of standards and for sample processing and preservation must be ultra-high purity grade or the equivalent. Redistilled acids are acceptable.

Nitric acid, concentrated (sp gr 1.41).

6.2 Deionized, distilled water—Prepare by passing distilled water through a mixed bed of cation and anion exchange resins. Use deionized, distilled water for the preparation of all reagents, calibration standards and as dilution water.

6.3 Standard stock solutions (1 mL containing 1000 µg of the element to be determined) may be purchased or prepared from ultra-high purity grade chemicals or metals. If available, materials traceable to NBS Standards should be used.* All salts must be dried for 1 h at 105 °C, unless otherwise specified, prior to preparation of stock solutions.

6.4 Two types of blanks are required for the analysis. The calibration blank (Section 3.9) is used in establishing the analytical curve. The reagent blank (Section 3.8) is used to correct for possible contamination resulting from varying amounts of acid used in the sample processing.

6.4.1 The calibration blank must contain the same concentration of nitric acid as that present in the standards used to define the calibration curve.

6.4.2 The reagent blank must contain nitric acid in the same volume as that used in the processing of the samples. The reagent blank must be carried through the complete procedure and contain the same acid concentration in the final solution as the sample solution used for analysis.

6.5 In addition to the calibration standards, an instrument check standard (Section 3.4) and a reference standard (Section 3.5) are also required for the analyses.

6.5.1 The instrument check standard is prepared by the analyst to contain elemental concentrations equivalent to the midpoints of the corresponding calibration curves. This standard should be included in the analytical scheme with a frequency of 10%.

6.5.2 The reference standard should be prepared according to the instructions provided by the supplier. Following initial verification of the calibration standards, analyze weekly.

## 7. Sample Handling and Preservation

7.1 For the determination of trace elements, contamination and loss are of prime concern. Dust in the laboratory environment, impurities in reagents, and impurities in laboratory apparatus which the sample makes contact with are all sources of potential contamination. Sample containers can introduce either positive or negative errors in the measurement of trace elements by (1) contributing contaminants through leaching or surface desorption and (2) by depleting concentrations through adsorption. Thus, the collection and treatment of the sample prior to analysis requires particular attention. Laboratory glassware including the sample bottle (whether linear polyethylene, polypropylene, or Teflon) should be thoroughly washed with detergent and tap water; rinsed with 1:1 nitric acid, tap water, 1:1 hydrochloric acid, tap water, and finally deionized, distilled water in that order.*

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* Caution: Many metal salts are extremely toxic and may be fatal if swallowed. Wash hands thoroughly after handling.

7.2 For the determination of total elements, known volumes of representative samples of liquid residual wastes should be acidified to pH <2 with concentrated nitric acid as soon as possible after collection. The volume of acid used for the pH adjustment may vary considerably for various types of industrial waste and may result in significant dilution of the sample. The volume of nitric acid used for preservation must be recorded to allow calculation of a dilution factor. Dry, solid residual wastes should be stored in clean, capped plastic or glass containers.

## 8. Sample preparation

8.1 For the determination of total elements, take an approximate volume (previously determined by a preliminary screening of the sample) of the liquid waste sample and transfer it to a clean pretared 50-mL glass ampul. Reweigh the ampul and record the weight of the sample. Add 2 mL of concentrated nitric acid, dilute to about 20 mL, if necessary, with deionized, distilled water, and gently warm the sample until any gas evolution subsides. Seal the tip of the ampul with a torch making certain that a leak-free seal is obtained. Place the ampul in a 125 °C oven and digest for 1 h. For solid wastes, accurately weigh out a sample, not to exceed approximately 5 g, and quantitatively transfer it to a clean 50-mL ampul. Add 20 mL of 10% nitric acid, warm until gas evolution subsides, and seal. Digest for 1 h at 125 °C. Cool the ampul, wrap with a towel, and snap off the tip.** All samples should be at pH <2 after digestion.

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* Chromic acid may be useful to remove organic deposits from glassware; however, the analyst should be cautioned that the glassware must be thoroughly rinsed with water to remove the last traces of chromium. This is especially important if chromium is to be included in the analytical scheme. A commercial product, NOCHROMIX, available from Godax Laboratories, 6 Varick St., New York, NY 10013, may be used in place of chromic acid. Chromic acid should not be used with plastic bottles.

If it can be documented through an active analytical quality control program using spiked samples and reagent blanks that certain steps in the cleaning procedure are not required for routine samples, those steps may be eliminated from the procedure.

** Caution: Significant pressure buildup may occur in the ampul during the digestion step; therefore, the analyst should wrap the ampul in a towel in case shattering should occur during opening.

If not, repeat the digestion using a sufficient volume of concentrated nitric acid to maintain acidic conditions. Quantitatively transfer samples containing no solid residue to a 50-mL volumetric flask with deionized, distilled water and dilute to volume. For samples containing solid residue, suction filter the sample through a prewashed Whatman No. 42, or equivalent, filter paper. Rinse the ampul several times with deionized, distilled water and filter the rinsings through the residue. Wash the residue with deionized, distilled water and dilute the filtrate to a final volume of 50 mL. The sample is now ready for the analysis of total metals.

## 9. Procedure

9.1 Set up the instrument with proper operating parameters established in Section 5.2 for direct aspiration, furnace, gaseous hydride, or cold vapor analysis. Allow at least 30 min for the instrument to stabilize prior to analysis.

9.2 Develop linear calibration curves for each element according to the instrument manufacturer's recommended procedures, using appropriate standards prepared from stock solutions described in Section 6.3.

9.3 Before beginning a sample run, reanalyze the reference standard (Section 6.5.2) to ensure that no changes in instrument sensitivity have occurred.

9.4 Begin the sample runs. For flame analysis, flush the system with the calibration blank (Section 6.4.1) between each sample. Analyze the reference standard (Section 6.5.2) every 10 samples.

9.5 If it has been found that methods of standard addition are required, the following procedure is recommended:

9.5.1 The standard addition technique involves preparing new standards in the sample matrix by adding known amounts of standard to one or more aliquots of the processed sample solution. This technique compensates for a sample constituent that enhances or depresses the analyte signal, thus producing a different slope from that of the calibration standards. It will not correct for additive interference, which causes a baseline shift. The simplest version of this technique is the single-addition method in which two identical aliquots of the same solution, each of volume $V_x$, are taken. To the first (labeled A) is added a small volume $V_s$ of a standard analyte solution of concentration $C_s$. To the second aliquot (labeled B) is added the same volume $V_x$ of the solvent. The analytical signals of A and B are measured and corrected for nonanalyte signals. The unknown sample concentration $C_x$ is calculated:

$$C_x = \frac{S_B V_s C_s}{(S_A - S_B)\, V_x}$$

where $S_A$ and $S_B$ are analytical signals (corrected for the blank) of solutions A and B respectively. $V_s$ and $C_s$ should be chosen so that $S_A$ is roughly twice $S_B$ on the average. It is best if $V_s$ is made much less than $V_x$ and thus $C_s$ is much greater than $C_x$ to avoid excess dilution of the sample matrix. If a separation or concentration step is used, the additions are best made first and carried through the entire procedure. For the results for this technique to be valid, the following limitations must be taken into consideration:

a. The analytical curve must be linear.

b. The chemical form of the analyte added must respond the same as the analyte in the sample.

c. The interference effect must be constant over the working range of concern.

d. The signal must be corrected for any additive interference.

## 10. Calculation

10.1 Analytical signals for reagent blanks (Section 6.4.2) should be subtracted from signals for all samples. This is particularly important for digested samples requiring large quantities of acids to complete the digestion.

10.2 If dilutions are performed, the appropriate factor must be applied to sample values.

10.3 Results should be reported in µg/g of undiluted residual waste. For concentrations below 1 µg/g, report as <1 µg/g; for concentrations above 1 µg/g, report up to three significant figures.

## 11. Quality Control (Instrumental)

11.1 Analyze the instrument check standard (Section 6.5.1) at a frequency of 10%. This check standard is used to determine instrument drift. If agreement is not within ±2% of the expected values or within the established control limits, the analysis is out of control.

11.2 For the purpose of verifying interelement and/or background correction factors, analyze a second check standard prepared by spiking a representative sample with the analytes of interest at either 10 times the detection limit for samples containing low concentrations of analyte, or 2 times the concentration for samples containing high concentrations of analyte. Values should fall within the established control levels of 1.5 times the check standard.

11.3 A reference standard (Section 6.5.2) from an outside source, but having known concentration values, should be analyzed as a blend sample on a weekly frequency. Values should be within the established quality control limits. If not, prepare new stock standards.

## 12. Quality Control (Method)

12.1 The precision of the analyses should be determined by analyzing a minimum of five replicates of each type of residual waste.

12.2 The accuracy of the method should be determined from the spike recovery data obtained by the analysis of a minimum of five replicates of the same residual waste, spiked prior to digestion of the sample, and carried through the same sample preparation method used in Section 12.1.

## 13. References

13.1 "Procedures of Analysis of Heavy Metals and Other Elements in Sewage or Sludge," University of Washington, Laboratories of Radiation Ecology Research Report, February 21, 1980.

# D. Quality Assurance and Quality Control Procedures for Determination of Purgeable Organics and Base/Neutral and Acid Extractable Organics in Residual Waste

# Quality Assurance and Quality Control Procedures for Determination of Purgeable Organics and Base/Neutral and Acid Extractable Organics in Residual Waste*

## 1. Scope and Application

1.1 These procedures are provided for use by laboratories performing analyses by the proposed methods for purgeable and semivolatile organics in residual waste. To provide data with a known degree of reliability, a strong Quality Assurance and Quality Control program is presented. The procedures are designed to produce data with known precision and accuracy so that a determination of confidence can be placed in the data. Quality Assurance (QA) is the total program for ensuring the reliability of the monitoring data. Quality Control (QC) is the routine application of procedures for controlling the measurement process.

1.2 The Quality Assurance plan includes the following elements:

1.2.1 Spiked reagent water samples.

1.2.2 Daily use of reference compounds to verify ion abundance measurements: decafluorotriphenylphosphine (DFTPP) for semivolatiles and p-bromofluorobenzene (BFB) for purgeables.

1.2.3 Daily use of a Quality Control sample to establish GC instrument performance: benzidine at the 100-ng level for base/neutrals and pentachlorophenol at the 250-ng level for acids.

1.2.4 Spike recovery and duplicate analyses to establish precision and accuracy.

1.2.5 Routine use of surrogate spikes for quality control and internal standards for quantitation.

1.2.6 Method blanks with every sample set.

1.3 Initially, the methodology must be validated for each industrial subcategory being measured by the laboratory. The requirement for validation of each subcategory is based on the assumed unique nature of the residual waste associated with each subcategory. The Quality Assurance data are used to define the performance of the specified methodology on the individual waste matrices.

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* The quality control and the quality assurance procedures for metals are similar to those for purgeable and semivolatile organics. For those instances where there are very specific differences, the quality control and quality assurance procedures are given in detail in the proposed method for metals in residual waste.

## 2. Quality Assurance Requirements

2.1 Field blank—The field blank is defined as an appropriate volume of reagent water that is sent to the sampling site and back to the analytical laboratory in a container and bottle identical to the type used to collect the samples. Field blanks and samples must be shipped in separate containers. An appropriate field blank is provided to the analyst by the sampling crew. When received in the laboratory, the field blank is spiked with surrogates, extracted, and concentrated as if it were an actual sample.

2.1.1 For the semivolatile organics (base/neutrals or acids), the minimum requirement is to provide an appropriate volume of blank water that has been processed through the sampling equipment in the same manner as a sample. The field blank is then analyzed in the laboratory as if it were a sample. The field blank may be screened by GC/FID. Full GC/MS analysis is to be performed if the screening analysis gives any peaks larger than the internal standard peak. When contamination of the field blank occurs, the analytical results must be discarded or flagged so as not to result in the reporting of false positives in the authentic samples.

2.1.2 Field blanks for the purgeable organics consists of "organic-free" water that is sent from the laboratory to the sampling site and retained with the samples. The purpose is to check on possible contamination of the sample by permeation of volatiles through the septum seal. Sample blanks should be protected during transit and in the laboratory by storage of the sample bottles in a sealed container with activated carbon.

2.2 Method blank—The method blank is defined as an appropriate volume of "organic-free" water that is processed exactly as a sample is processed (same glassware, reagents, solvents, etc.). A method blank must be processed for each set of field samples extracted at a given time (at least one method blank per 20 field samples analyzed) and whenever a new source of reagent or solvent is introduced into the analytical scheme. The method blank can be screened by the GC/FID. Analysis by GC/MS is required only if GC/FID analysis of the field blank gives any peaks larger than the internal standard peak. Reagents or solvents having background levels that interfere with the compounds to be determined must be purified and shown to be acceptable or replaced with some that are acceptable before proceeding with the analyses. Problems encountered and corrective actions taken shall be documented and reported for the record.

2.2.1 For the extractable semivolatile organics (base/neutrals or acids), the method blank requires extraction of 40 mL of reagent water.

2.2.2 For the purgeable organics, 10 mL of reagent water should be analyzed by the purge-and-trap methodology only if positive interferences are noted during the analysis of a field blank. If positive interferences still occur, repeat the method blank analysis. If interference persists, dismantle the system, thoroughly clean all parts that make contact with the sample, purge gas, and carrier gas. Replace or repack the sorbent trap and change the purge and carrier gas.

2.3 Analytical standards—The proposed methods for purgeable and semivolatile organics in residual waste assume the preparation of stock solutions from pure, authentic samples of the compounds to be determined. Commercially available mixed stock solutions, if they are shown to be satisfactory, may be used to prepare standard solutions and spiked samples. Reagents and standards should carry a well-affixed label providing identification and concentration, preparation date, name of responsible person and identification numbers that allow the solutions to be traced to the analytical records for their preparation. A minimum three-point calibration curve should be prepared for each compound determined. The calibration curve should bracket the concentration for that particular compound in the sample to be analyzed. Standards should be kept refrigerated in serum bottles with Teflon-lined caps and the liquid level marked on the bottles. Stock solutions of most standard compounds that boil above room temperature are generally stable for at least four weeks when stored at 4 °C; however, the stability should be checked on a periodic schedule.

2.4 Surrogate spikes—Surrogate spikes are defined as standards that are added to every sample prior to sample processing and analysis. The standards chosen should be chemically similar to compounds in the fraction being analyzed. Also, the standards should be compounds that would not likely be found in environmental samples. The purpose of the surrogate spikes is to provide quality control on every sample by constantly monitoring for unusual matrix effects, gross sample processing error, etc. The surrogate spikes should not be used as internal standards for quantitation purposes.

2.5 Internal standards—Internal standards are defined as compounds that are added to the sample no more than a few minutes before its injection into the GC/MS to minimize the possibility of losses due to evaporation, adsorption, or chemical reaction. The standard compounds chosen should be chemically similar to compounds in the fraction being analyzed. Compounds used as internal standards are not to be used as surrogate standards. The internal standards must be different from the surrogate standards.

2.6 Replicates—To determine the precision of the method, a regular program of analyses of replicate aliquots of environmental samples must be carried out. At least three replicate aliquots of a well-mixed sample must be analyzed with each set of 20 samples or less from an industrial subcategory that is analyzed at a given time. For those compounds where a sufficient number of positive results are accumulated over a period of time, precision criteria should be developed. A minimum of 15 replicates at a particular concentration or concentration range where linearity exists is required to start an on-going program for QA and subsequent estimates of precision.

2.7 Spiked samples—These are samples that contain additions of known amounts of authentic analytical standards. These samples are processed and analyzed in the same manner as a sample (including addition of surrogate and internal standard solutions). The spiked samples are analyzed along with each set of 20 samples or less from each industrial subcategory. Spike data are obtained for each compound to be determined.

2.8 Spiked method blanks—These are obtained by additions of known amounts of authentic standards to the water blank before analysis. These blanks are then processed and analyzed in the same manner as a sample. The standards should be present at approximately the concentrations found in routine samples. The percent recoveries are determined as described later in this protocol. At least two spiked method blanks must be analyzed along with each set of 20 samples or less.

2.9 GC performance evaluation.

2.9.1 For the base/neutral semivolatile organics, 100 ng of benzidine is run daily either separately or as part of a standard mixture that may also contain 50 ng of DFTPP. The tailing factor for benzidine should be less than three. Calculation of this factor is described in Figure V-10.

2.9.2 For the acid semivolatile organics, 250 ng of pentachlorophenol is run daily either separately or as part of a standard mixture that may also contain 50 ng of DFTPP. The tailing factor for pentachlorophenol should be less than five. Calculation of this factor is described in Figure V-10.

2.10 GC/MS calibration evaluation.

2.10.1 For the extractable semivolatile organics, 50 ng of decafluorotriphenylphosphine (DFTPP) is run daily and checked to ensure that the performance criteria listed in Table V-11 are met. If the system performance criteria are not met, the analyst must retune the mass spectrometer and repeat the performance evaluation. The performance criteria must be met before any samples or standards may be analyzed.

2.10.2 For the purgeable organics, 100 ng of p-bromofluorobenzene (BFB) is run daily and checked to ensure that the performance criteria listed in Table V-11 are met. If the system performance criteria are not met, the analyst must retune the mass spectrometer and repeat the performance evaluation. The performance criteria must be met before any samples or standards may be analyzed.

## 3. Mathematical Calculations

3.1 Precision—For each compound, use the concentrations of compounds in the samples or the spiked samples ($0_1, 0_2, 0_3, \ldots, 0_n$) to calculate the standard deviation (S) of replicate analyses according to Equation 1.

$$S = \sqrt{\frac{n \sum_{i=1}^{n} 0_i^2 - \left( \sum_{i=1}^{n} 0_i \right)^2}{n(n-1)}} \qquad (1)$$

3.2 Accuracy.

3.2.1 For each compound, use the concentration values for the samples or the spiked samples ($0_1, 0_2, 0_3, \ldots, 0_n$) to calculate the mean value of replicate analyses ($\bar{X}$) according to Equation 2.

$$\bar{X} = \frac{\sum_{i=1}^{n} 0_i}{n} \qquad (2)$$

where n = number of replicates.

3.2.2 For each compound, use the resulting mean concentration values of the samples and of the spiked samples to calculate the mean percent recovery ($\overline{P}$) of the method according to Equation 3.

$$\overline{P} = \frac{\overline{X}_L - \overline{X}_0}{T} \qquad (3)$$

where $\overline{X}_L$ = mean observed concentration of replicates of the spiked sample at a given spike level.

$\overline{X}_0$ = mean observed concentration of replicates of the sample not spiked.

T = true value or calculated concentration of the spike added to the sample.

3.3 The precision and accuracy data shall be documented for the record as evidence that the laboratory can properly process the samples and perform chromatography essential for the proposed methods for purgeable and semivolatile organics.

## 4. Preliminary Precision and Accuracy with Compounds in Clean Water

4.1 Before any work is begun on actual field samples, a laboratory must demonstrate its ability to perform properly the liquid-liquid extractions, the gas purge extractions, and the required chromatography. Clean water spikes are analyzed to demonstrate the laboratory's ability to implement the proposed methods for purgeable and semivolatile organics.

4.2 Prepare reagent water for use in determining preliminary precision and accuracy according to the procedure in the proposed methods for purgeable and semivolatile organics.

4.3 Spike four replicates of reagent water with an authentic sample of each compound to be determined at a concentration approximately equal to 10 times the limit of detection. In addition, spike all aliquots with a minimum of three surrogate standards. For semivolatile organics, each replicate must be 40 mL; each purgeable sample requires at least 10 mL (do not dose purgeables with more than 2 μL of an alcoholic standard per each 10 mL of water). Analyze the spiked solutions according to the proposed methods for purgeable and semivolatile organics.

4.4 Determine precision and accuracy according to Section 3.

## 5. Method Validation

5.1 Semivolatile organics—The following procedures are to be applied, separately, to samples being analyzed for the base/neutral and acid groups of compounds. The analysis shall be performed according to the procedures given in the proposed method for these classes of organics. The validation studies must be performed under the same conditions ordinarily applied to the samples of a given subcategory.

5.1.1 Sample pretreatment—The laboratory should collect a sample of adequate volume to carry out the validation study described here and should collect one field blank by the method described in the sampling protocol. Mix the sample with some type of stirring device. Withdraw aliquots, while stirring, and add them to a 250-mL round-bottom centrifuge bottle, using a siphon made of glass or Teflon. Measure and record the aliquot weights precisely. Initially analyze the 40-g aliquots to determine the sample background so that proper spiking levels can be selected for Section 5.1.2. The remainder of the sample should be stored at 4 °C until the validation study is begun. At the same time that the sample aliquot is analyzed, analyze a 40-g aliquot of the field blank.

5.1.2 Spiking of aliquots—All sample aliquots are spiked with surrogate standards. Five aliquots for each spiking level are spiked with surrogate standards plus the standard pollutant compounds of interest. Select the three spiking levels (concentrations) for the compounds of interest based on the results of the background analysis obtained in Section 5.1.1. If the initial background level for a particular pollutant is X, select the spike levels to give final concentrations of 2X, 10X, and 100X. If X equals 20 μg/g, spike with 20, 180, and 1980 μg/g. This gives final concentrations of the pollutant of 40, 200, and 2000 μg/g. Spike each 40-g replicate with each surrogate at a level of 20 μg/g.

5.1.3 Prepare spiking standards in concentrations such that no more than 1 mL of spiking solution is added for each 40 g of sample. This will insure that the solubility of the standard in water will not be significantly affected by the added organic solvent. Add the spiking solution to the sample aliquots in the round-bottom centrifuge bottle by use of a transfer pipet or microsyringe. After adding the spikes, thoroughly mix the samples and after 1 h at room temperature proceed with the extraction.

5.1.4 Use of the data from spiked samples in analyses—The data obtained from the determination of semivolatile organics of interest are used to calculate the precision and accuracy of the method and to establish control limits for the individual compounds of interest. The addition of surrogate spikes provides a quality control on every sample by monitoring for matrix effects and gross sample processing errors. The surrogate is not used as an internal standard for quantification purposes. Suggested surrogate standards are 2-fluorobiphenyl, 2,2'-difluorobiphenyl, and 2-fluoronaphthalene for base/neutral compounds and 2-fluorophenol, pentafluorophenol and α,α,α-trifluoro-m-cresol for the acid compounds.

5.1.5 Internal standards are added to the concentrated sample prior to GC/MS analysis to provide quality control on every sample by monitoring the possibility of losses due to changes in operating parameters. Internal standards are used for quantification purposes. Suggested internal standards are naphthalene-$d_8$, anthracene-$d_{10}$, and nitrobenzene-$d_5$ for the base neutral compounds and phenol-$d_6$ for the acid compounds.

5.2 Purgeable organics.

5.2.1 The validation of the method for purgeables requires a minimum of 500 g of sample. The validation may be performed on a grab sample or a composite sample prepared from discrete grab samples.

Thirteen aliquots of each sample are required. They should be treated and spiked according to Sections 5.2.2 d through 5.2.2 e and 5.2.6. The remaining sample is transferred to a clean vial and sealed with no headspace, as is done when the sample is first collected. This sample should be held at 4 °C until the determination is made that there is no further need for the sample.

Caution: Prepare only as many sample aliquots as can be analyzed in the working day. This may mean that each of the three concentration levels will be analyzed on different days.

5.2.2 Pretreatment of grab samples to be composited—Individual grab samples should be composited according to the following procedure:

a. Composite only grab samples of equal volume.

b. Carefully pour the contents of all individual grab samples collected from a given source during the specified time period into a 1000-mL wide-mouth flask, which is chilled in a wet ice bath.

c. Stir the mixture gently with a glass rod for approximately 1 min while the mixture is in the ice bath.

d. Carefully fill 13 clean 40-mL vials, or three 120-mL vials and four 40-mL vials, with the composited sample.

e. Store the vials at 4 °C until the validation study is begun.

5.2.3 Spiking levels for analytical standards and surrogate standards—The spiking levels of the analytical standards are determined by the background (X) in the sample. The low level spike will give a final concentration that is 2 times the background level. The intermediate and high level spikes will give final concentrations that are 10 and 100 times the background level. Concentrations in excess of 1 μg/g are likely to exceed the linear range of the method. Therefore, the total concentration (background plus spike) of each individual compound should not exceed 1 μg/g. Even at this level, the solubility of the compounds in the sample must be considered. The spiking level for all surrogate standards should be 1 μg/sample aliquot.

5.2.4 Preparation of spiking standards—Prepare methanolic stock solutions of the analytical standards and the surrogate standards according to the directions given in the proposed method for purgeable organics in residual wastes.

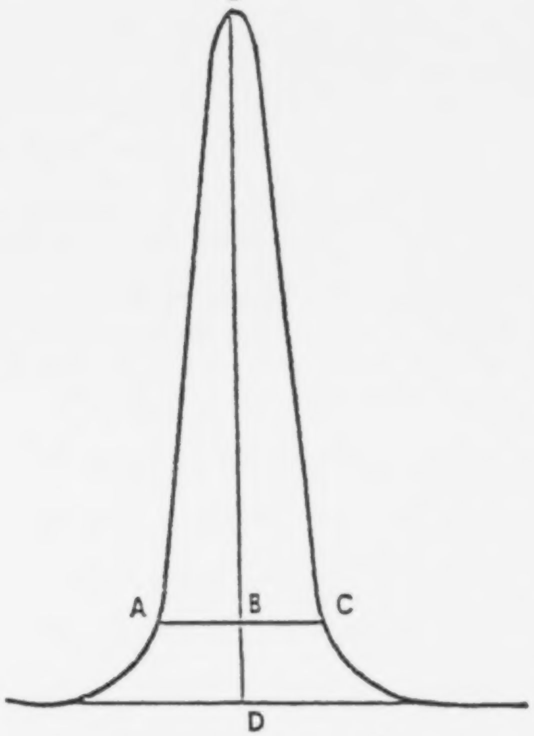
5.2.5 Take one 40-mL vial for immediate analysis to determine the background of the purgeable. Weigh an appropriate sample into a pretared 10- to 15-mL Teflon-lined, screw-capped vial. Dilute the sample to 10 mL with reagent water. Transfer the total sample or an aliquot to the purge device using a syringe with a 1/8-in. gauge Teflon needle. Add an appropriate volume of surrogate and internal standard solutions. Seal the sample in the purge device. Analyze the sample aliquot according to the proposed method for purgeable organics in residual waste.

5.2.6 Spiking the sample aliquots—Weigh an appropriate sample into a pretared 10- to 15-mL Teflon-lined, screw-capped vial. Add an appropriate volume of solution of the authentic compound to be determined. Mix and proceed as in Section 5.2.5.

5.2.7 Calculation of precision and accuracy—The precision and accuracy of the purgeable organics and the surrogate standards are calculated as directed for the extractable organics in Section 3.

## 6. Reference

6.1 Appendix III—Example Quality Assurance and Quality Control Procedures for Organic Priority Pollutants, FR 44 (233), 69553-69559, 1979).



TAILING FACTOR = $\frac{BC}{AB}$

Example calculation: Peak Height = DE = 100 mm
10% Peak Height = BD = 10 mm
Peak Width at 10% Peak Height = AC = 23 mm
AB = 11 mm
BC = 12 mm

Therefore: Tailing Factor = $\frac{12}{11}$ = 1.1

Figure V-10. Tailing factor calculation

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TABLE V-11. IONS AND ION ABUNDANCE CRITERIA OF DECAFLUOROTRIPHENYLPHOSPHINE (DFTPP)

| M/E | Ion abundance criteria |
|---|---|
| 51 | 30 to 60% of mass 198 |
| 68 | Less than 2% of mass 69 |
| 70 | Less than 2% of mass 69 |
| 127 | 40 to 60% of mass 198 |
| 197 | Less than 1% of mass 198 |
| 198 | Base peak, 100% relative abundance |
| 199 | 5 to 9% of mass 198 |
| 275 | 10 to 30% of mass 198 |
| 365 | Greater than 1% of mass 198 |
| 441 | Present but less than mass 443 |
| 442 | Greater than 40% of mass 198 |
| 443 | 17 to 23% of mass 442 |